AD NUMBER

AD402993

NEW LIMITATION CHANGE

TO

Approved for public release, distribution unlimited

FROM

Distribution authorized to U.S. Gov't. agencies and their contractors; Administrative/Operational Use; 19 MAR 1963. Other requests shall be referred to Bureau of Naval Weapons, Washington, DC 20350.

AUTHORITY

USNASC ltr, 28 May 1971

PRODUCTION OF THIN POLYTETRAFLUOROETHYLENE RESIN
(TEFLON) COATINGS BY ELECTRODEPOSITION METHODS

By
Robert W. Logan

Contract No. Now-62-0600-C
Bureau of Naval Weapons, Department of the Navy

Final Report

Report Period: March 19, 1962
March 19, 1963

General Plastics Corporation

55 LA FRANCE AVENUE, BLOOMFIELD, NEW JERSEY

for BuWeps

PRODUCTION OF THIN POLYTETRAFLUOROETHYLENE RESIN
(TEFLON) COATINGS BY ELECTRODEPOSITION METHODS

By
Robert W. Logan

Final Report

Report Period: March 19, 1962
March 19, 1963

Submitted by: _Robert W. Logan_
Robert W. Logan

GENERAL PLASTICS CORPORATION
Bloomfield, N. J.

Robert Goldsmith, President

NOTICE

This document may not be reproduced or published in any form in whole or in part without prior approval of the Bureau of Naval Weapons, Department of the Navy.

Foreign announcement and dissemination of this report by DDC not authorized.

FOREWARD

This is the final report for the period of 19 March, 1962 to 19 March, 1963 prepared by General Plastics Corporation, Bloomfield, New Jersey, under Bureau of Naval Weapons Contract No. Now-62-0600-C.

ACKNOWLEDGMENTS

GENERAL PLASTICS CORPORATION

TABLE OF CONTENTS

SUMMARY

An electrodeposition method of applying thin polytetrafluoroethylene resin ("Teflon") coatings has been developed which allows the application of crack-free films up to 0.001" thick in one coating operation. Polytetrafluoroethylene (TFE) coatings applied by this method are intended to supplement sprayed TFE coatings in lubricating, without the use of oils or greases, a wide variety of military equipment.

Electrodeposited TFE coatings possess a very low coefficient of friction on steel, are smooth and free from blisters, cracks, coagulated particles and other surface defects and exhibit adhesion (under simulated dry lubricant conditions) comparable to sprayed TFE coatings.

Electrodeposited TFE coatings are plated from a bath composed of TFE (Teflon 41BX), perfluorooctanoic acid (stabilizer), pigment and fluorinated ethylene propylene (FEP) or alumina modified colloidal silica as adhesion aids. Extending the sintering time over that normally employed, aids in improving the adhesion of the electrodeposits to steel substrates.

The electrodeposition process will allow the coating of parts or equipment that heretofore was difficult, if not impossible, to spray. In addition, due to the generally favorable edge in the economics of the electrodeposition process vs. the spray process, TFE may now be applied to parts which previously were not economically feasible to coat.

The main limitation of the electrodeposition process is that only those metals which are anodically corrodible and which can withstand the 700°-750°F. sintering temperature can be coated. This eliminates aluminum, stainless steel and zinc, among others.

In spite of its limitations, the electrodeposition method of applying thin coatings of TFE should prove a valuable adjunct to the spray method of application.

I. INTRODUCTION

A. Development of Thin Teflon Films as Lubricant and Preservative Coatings for Metals.

Polytetrafluoroethylene (DuPont "Teflon") is noted for its extremely low coefficient of friction. Under certain test conditions, coefficients as low as 0.02 have been measured (1). As the static coefficient is approximately equal to the dynamic value, Teflon does not exhibit a stick-slip type of motion. This property, and the fact that the other properties of TFE are retained over a wide temperature range, makes Teflon attractive for dry lubricant applications. However, Teflon is a relatively soft material (Durometer D55-70) which causes it to be deformed rather easily and, due to its low thermal conductivity (2) heat is not dissipated readily from it. Because of these factors the use of bulk Teflon as a dry lubricant is limited to low speed, low load applications.

Fitzsimmons and Zisman of the Naval Research Laboratory discovered that by employing thin coatings of Teflon (TFE) backed up by a hard metal substrate, higher speeds and loads could be tolerated (3). Following their extensive and complete investigation of the lubricant and preservative properties of Teflon coatings they determined that for optimum lubricity, coupled with good adhesion, resistance to deformation, and corrosion resistance, the coating thickness should be in the range of 0.0006-0.0010 inches. In their report they describe a number of successful dry lubricant applications of thin TFE coatings to U.S. Navy and U.S. Marine Corps equipment.

Thin coatings of TFE are presently applied to metal parts by spraying a water dispersion of colloidal TFE particles (propriety formulations of the Finishes Division of the DuPont Company) onto the metal surface and subsequently drying and sintering at 700°F.-750°F. to coalesce the TFE particles. A two coat system is normally required to obtain the recommended 0.0006"-0.0010" thickness. Dip coating and flow coating techniques find only very limited application. Spray application, however, has a number of disadvantages. From an economy standpoint it is not attractive due to the considerable loss of coating material through "overspray". Because the final coating thickness must normally be attained in two or three applications in order to prevent "mud cracks" or obtain adequate adhesion, material and labor costs are further increased. The necessity for sintering each individual coat at 700°F.-750°F. increases overhead costs. Labor costs are further increased due to the considerable hand labor required to remove or level coagulated TFE particles which tend to develop in the high shear areas of the spray gun and be deposited on the work. Although the spray method of application is quite flexible there are a number of odd shapes and sizes of equipment and parts which are more amenable to coating by other methods.

It is the object of this study to eliminate or lessen the above disadvantages of the spraying process and to broaden the range of parts and equipment that may be properly TFE coated by developing the electrodeposition (or more properly electrophoretic) method of application to a commercially feasible process.

B. Theory of Electrodeposition from Non-metallic Colloidal Dispersions.

Many resins, elastomers and other materials, exist or can be manufactured in the form of colloidal sized particles dispersed in an aqueous or organic medium. The colloidal particles, when dispersed in aqueous medium, possess a tremendous surface area and normally acquire an electrical charge. When an anode and cathode are placed in these dispersions and a unidirectional electromotive force (emf) applied, the colloidal particles are displaced toward one of the electrodes, depending on the charge on the particles. In the immediate vicinity of this electrode the charge on the particles is neutralized and the particles coagulated into a wet compact deposit around the electrode. Although electrodeposition of colloidal particles dispersed in organic medium is possible, water is the preferred medium. Since most synthetic or naturally ocurring substances possess a negative charge in aqueous medium, deposition takes place at the anode. This process is called "anode electrodeposition". The origin of the negative charge on the colloidal particles is due to the adsorption of anionic surfactants and/or adsorption of hydroxyl (OH^-) ions. The electrodeposition of colloidal particles dispersed in water onto a metal substrate (the anode) is the result of four physical and chemical phenomena taking place simultaneously, namely: electrophoresis, electroosmosis, electrolysis, and electrochemical coagulation.

Electrophoresis is the movement of the charged colloidal particles toward the anode. This results in an increase in the concentration of colloidal particles in the immediate vicinity of the anode. The mobility of the particles is related to the voltage, dielectric constant, and viscosity by the equation:

$$V = \frac{Z \, E \, D}{4 \, \pi \, n}$$

where

  V = Velocity of the particles
  Z = Charge of the particle
  E = Voltage
  D = Dielectric constant of the suspending medium
  n = viscosity

Electroosmosis is the movement of positively charged water molecules or hydrated cations toward the cathode. This results in a further compacting of the deposit around the anode.

Electrolysis is concerned with the reactions taking place at the electrodes and involving a gain or loss of electrons. The main reactions are the electrolysis of water resulting in the formation of hydrogen gas at the cathode,

$$2H^+ + 2e \longrightarrow H_2\uparrow$$
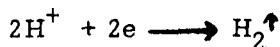
and, perhaps, oxygen evolution at the anode.

$$4OH^- \; -4e \longrightarrow O_2 + 2H_2O$$
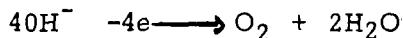
Oxygen evolution at the anode (or work) causes blisters, pits and other defects in the coating if not prevented. Hydrogen evolution at the cathode does not normally effect the quality of the coating being deposited at the anode. If the metal anode (such as steel) is sufficiently activated, electrochemical dissolution occurs forming ferrous ions (Fe^{++})

$$Fe^0 \quad -2e \longrightarrow Fe^{++}$$
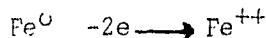
which move out toward the cathode.

Electrochemical coagulation is the neutralization of the negative charges on the colloidal particles caused by collisions with H^+ or Fe^{++} (if steel is the anode material). The Fe^{++} also react with OH^- to form metal hydroxides of hydrous oxides which may then be present in the deposits in small amounts.

$$2Fe^{++} + 4OH^- \longrightarrow 2Fe(OH)_2$$
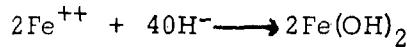
This reaction tends to prevent the liberation of oxygen gas on the anode (the work).

The wet compact deposit thus formed (an insulator when dry) contains sufficient occluded water to maintain electrical conductivity and permit the deposition to continue. As the solids content of the deposit being formed is higher than the solids content of the bath, the solids content of the bath will decrease. Due to the discharge of H^+ at the cathode, an excess of OH^- generally develops which results in an increase in the alkalinity of the bath as deposition continues.

C. History of Electrodeposition from Non-metallic Colloidal Dispersions.

Although the phenomena of electrophoresis (the movement of charged particles in an electric field) was known many years before, the first application of the principle for coating metals with non-metallic substances was noted by Davey (4) who used electrodeposition methods to apply Japan. In the 1920's and early 1930's extensive studies were conducted on the electrodeposition of rubber latex. Several excellent articles and patents by the chief investigators in this field, such as Klein, Sheppard, Beal, Eberlin, and Szegvari appear in the literature (5,6,7,8,9). Several books on rubber latex contain excellent bibliographies on this subject (10,11). Although the process had rather widespread usage during this period, with the introduction of more concentrated rubber latices the electrodeposition process lost favor to the dipping process.

The literature also contains references to the use of electrodeposition methods for the application of other resins, such as vinylidene chloride, Linden and Townsend (12), vinyl chloride, Feinleib (13), nylon, Logan and Nelson (14), polyethylene, Gray (15), and cellulosics, Eberlin and Beal (16).

Electrodeposition of polytetrafluoroethylene (Teflon) is also noted. Hochberg (17) patented mixtures of Teflon with various film-forming additives to increase the "critical cracking thickness" of Teflon dispersions. Employing several of these film-forming additives in combination with Teflon, Heller (18) patented the electrodeposition of Teflon which is further described in a Du Pont Information Bulletin (19). The use of this process in the coating of wire and complex shapes is described (20, 21).

In spite of the many references in the literature to elecrophoretic methods of deposition, little commercial use of this method has been realized. This is due to one or more of the following reasons:

1. The electrolysis of water occurring simultaneously with the deposition of the colloidal particles can lead to porosity in the coating if oxygen evolution at the anode is not prevented.

2. The pH and the solids concentration of the bath change slightly with each plating cycle, thereby affecting the plating rate and efficiency and thus presenting a thickness control problem.

3. The possible priming techniques or additives that can be employed is limited by the fact that the coating must be applied in one application.

4. The number of substrates that may be coated is limited by the fact that some of the plastic or elastomeric coatings are not compatible with the ions which are developed from the metal surface.

5. The number of substrates that may be coated is further limited by the fact that the substrate must be capable of corroding anodically.

6. The principal intended use of this method was for the production of thick, corrosion resistant coatings, an area where production of 100% pore-free coatings in one application is difficult to achieve by any method.

7. Many of the coating materials applicable to this method of deposition can also be deposited easily by dipping methods or ionic deposition methods.

All previous research on electrodeposited TFE coatings was directed toward the production of heavy films. When considering the production of only thin TFE coatings, many of the above difficulties are eliminated or their effect greatly minimized. While true that the principal advantage of the electrodeposition method over other methods (i.e. the rapid rate of film build-up) is lost, the inability to produce acceptable TFE coatings by dipping methods and the problems and high costs associated with hand spray methods makes an investigation of the electrodeposition method of producing thin TFE coatings a worthwhile study.

D. Requirements of Electrodeposited TFE Coatings.

In their investigation of the use of TFE coatings as dry lubricant films, Fitzsimmons and Zisman determined that the best balance of frictional properties and durability is obtained when the final applied thickness is between 0.0006" - 0.001". The coatings should be well adhered to the substrate and be free of blisters, pits, coagulated particles, etc. The coatings should also be free of adulterants that would affect the inherent properties of TFE, such as the low coefficient of friction and the low wettability by water and oil. The above requirements are met by applying several coats of aqueous TFE dispersion (proprietary formulations of the Finishes Division of the Du Pont Company) by hand-spraying methods.

As the intended use is identical, the requirements of electrodeposited TFE coatings are the same as for hand-sprayed coatings. In order to meet these requirements, however, it was realized from previous electrodeposition experience with heavy TFE coatings that two main problems would have to be solved. First, the adhesion of unadulterated TFE coating materials, which are abhesive, would have to be increased as the adulterated TFE coating materials used to develop adhesion in the spraying process are not usable in the electrodeposition process. Second, the "critical cracking thickness" (or CCT) of the TFE plating materials would have to be increased to over 0.001" if the coatings are to be crack-free in the required range of 0.0006" - 0.001". Since once the wet deposit is dried it becomes an electrical insulator and the final coating thickness cannot be obtained by repeated applications as is true with the spray method of application. The CCT exhibited by

most TFE dispersions is approximately 0.0005", a value below the required coating thickness range.

It was realized early in this study that in addition to the need for increased adhesion and CCT of the coatings, the plating bath should exhibit the following desirable characteristics:

Good drainage properties – As the fresh wet electrodeposits are very soft and tender they must be removed from the bath slowly. The final appearance of the coatings depends to a degree on how well they drain during their removal, i.e. whether or not they tend to concentrate in rivers, retract, crawl, etc. These properties are mainly functions of the bath properties. To lower the withdrawal time it is desirable that the coatings drain quickly as the withdrawal rate must usually be slower than the drainage rate.

Poor foam stability – As the fresh coatings cannot be rinsed, it is desirable that the surface of the bath be completely free of foam. Otherwise bubbles may impinge on the work as it is removed, resulting in defects in the coatings. As it is impossible to prevent the development of some foam in the bath., it is desirable that the bath possess poor foam stability so that any foam produced may be easily collapsed by the action of a heated air blast.

Good bath stability – As it is expected that the electroplating bath will be in use for a long period of time before it is expended, the bath must possess good stability to coagulation and flocculation. Any settling which does occur should be of the "soft" variety so that the solids may be easily redispersed.

Good throwing power – As it is desired to coat complex shaped articles by this process, it is desirable that the bath exhibit the best possible throwing power, resulting in more even coatings and making unnecessary in most cases the construction of auxiliary cathodes.

E. Some Observations – Metallic Plating vs. Non-metallic Plating

The general procedure and set-up for producing non-metallic electrodeposits is similar to that for producing metallic deposits (the common electroplating process). The term "metallic electrodeposition" refers to the process wherein the metal to be electrodeposited is put into water solution (through ionization of one of its salts) and these ions on application of an electromotive force (emf) deposit at the cathode. A pure metal common to the positive metallic ion in solution serves as the anode. For satisfactory coatings to be produced by both methods, the substrate must be extremely clean.

The primary difference between the two methods is that in one case we are electrodepositing an electrical insulator, in the other a conductor. In non-metallic electrodeposition the conductivity of the deposit is maintained by the occluded water contained in the wet deposit being formed. In metallic electrodeposition, the deposit is itself an electrical conductor.

Non-metallic electrodeposition generally requires high voltage, although the current density is low due to the high resistance of the plating bath and may drop off in time due to the increased resistance of the deposit being formed. Metallic electrodeposition is normally conducted at low voltage but employs high amperage due to the high conductivity of the solutions.

5.

Non-metallic electrodeposition is normally conducted at room temperature with little or no agitation of the bath, whereas metallic electrodeposition is conducted at temperature ranges of 120° F.-180° F. employing considerable agitation, both factors allowing for higher current density without "burning" of the deposit.

During the non-metallic electrodeposition process the bath concentration normally will decrease due to the fact that the percentage of water contained in the wet deposit is lower than that contained in the bath.

In non-metallic electrodeposition the bath concentration is maintained by the periodic addition of a more concentrated dispersion, or by separating the anode and cathode compartments with diaphragms having electroendosmotic character. The bath concentration in metallic electrodeposition is maintained by the continuous dissolution of a common metal anode which dissolves at a rate approximating the rate at which the metallic ions are being "plated out".

Non-metallic deposits are soft and tender and contain considerable water which must be removed by slow drying. To coalesce the dried particles the coating must be sintered or baked. Metallic deposits are essentially complete once the metallic ions have been deposited on the work. They do, however, require rinsing and drying to eliminate staining.

Non-metallic electrodeposits are generally produced on the anode, whereas metallic electrodeposits are produced on the cathode.

Since the ratio of charge to particle size for non-metallic colloidal particles is much smaller than for metallic ions, the rate of deposition for non-metallic materials is much greater. When compared in terms of the weight of material deposited per faraday (96,500 ampere-seconds), non-metallic electrodeposition takes place on the order of 1,000 times faster. When compared in terms of the thickness produced per faraday, the difference is even greater due to the lower density of the non-metallic deposits.

F. Some Observations - Non-Metallic Plating (Heavy Coatings vs. Thin Coatings)

Early in this investigation it was realized that the relative importance of some of the plating parameters was dependent on whether thin or heavy films were being deposited. Unlike heavy deposits, which are considerably tough in the wet state, the thin deposits are generally not sufficiently tough to be rinsed following their withdrawal from the plating bath. Therefore, it is evident that any coagulum or gas picked upon the fresh deposit during withdrawal will lead to defects in the final coating. Although the toughest films are produced from baths containing rubber-like tacky additives, even unmodified TFE dispersions become tougher with increasing film thickness, although, of course, they mud crack on drying. When plating thin films, evidently the time element of deposition is so short (approximately 1-1/2 seconds) that the phenomena of electrophoresis or electroosmosis do not have sufficient time to come into play. Both of these phenomena serve to toughen the wet deposits. The principal reaction therefore appears to be one of electrochemical coagulation, or the coatulation of the TFE particles by the Fe^{++} ions developed at the anode.

Unlike the deposition of heavy coatings which generally experience some difficulty with simultaneous oxygen evolution at the anode via electrolysis of water, little difficulty is experienced when plating thin films. This is evidently due to the short time cycle involved.

6.

As the fresh thin deposits are considerably softer than the heavier films, it is necessary to withdraw them from the bath at a slow rate in order to form smooth coatings free from drainage marks. As the deposits are easily removed it is important that the surface of the bath be absolutely free of any foam, coagulum, or other debris. With heavy deposits this is not necessary. They are generally tough enough to allow rinsing off of any coagulum, etc. which may attach to the coating on withdrawal.

The times involved for plating of thin films of one-mil or less are on the order of 0.5 - 3.0 seconds, whereas heavier deposits require plating times of 1 - 15 minutes. Therefore, the plating of thin films can present a problem in the accurate control of thickness.

When plating only thin films it is essential that the viscosity and thixotropic properties of the bath be kept low. This requirement is necessary to prevent the pickup of a proportionally large amount of material during the withdrawal operation.

The most important difference in the plating of thin films vs. heavy films is the requirement for an extremely "active" surface. To obtain the degree of activity required, the freshly sandblasted piece must also be treated with an acid to further activate it prior to plating. If this step is eliminated it was noted that heavy coatings would be produced on the outer edges of the panel before any coating, or very little coating, was deposited on the middle areas of the panel. As the plating cycle continues, these middle areas, or initially passive areas, would become more active as the outer edges became insulated and more material would be deposited in the interior. However, by the time this point was reached, the outer edges would be many times over the thickness of the middle areas and average thicknesses on the order of 3 - 4 mil would have to be reached before the differences in outer and inner areas would be minimized.

In addition to the requirement for having the panel in a very active condition, it is necessary that the bath possess greater throwing power than is normally required in the plating of heavy films. The reason for this is apparently the same as the reason for requiring an active surface. In other words, as the plating takes place almost instantaneously, the deposit must be produced equally on all portions of the panel rather than the normal method of producing coatings on the high current density areas first and then on the lower current density areas as the higher current density areas become insulated.

Normal TFE dispersions possess a CCT of approximately .5 mil. In order to produce heavy coatings that are crack-free, it is necessary to add rubber-like elastomers such as Buna N. in amounts of approximately 30% of the TFE weight and a stepwise sintering cycle is required to remove the additive slowly to prevent blistering. The resulting coating, therefore, contains an appreciable quantity of carbonaceous material or is considerably porous. While not necessarily a major defect in the production of heavy films, it would be in the production of thin films. Therefore, since the requirement calls for a film thickness of .0006" - .001", techniques other than the addition of volatile film-formers are necessary to increase the CCT of the TFE plating baths.

G. Plating Parameters - Generalizations on Their Effect on the Quality and Rate of Deposition.

Generalizations as to the effect of various plating parameters on the quality of thin TFE deposits are discussed below.

1. Substrate

a. Cleanliness – Cleanliness of the metal is very important in any electro-deposition process. As all work is sandblasted to a white finish, a clean surface is assured.

b. Surface roughness – Since no levelling action takes place with thin TFE deposits, the surface roughness of the substrate will determine the roughness of the coated surface which, in turn, influences the lubricant and preservative properties of the coating. The Naval Research Laboratory has previously determined that for the correct balance of properties, the substrate should be sandblasted with #80 silica sand at air pressures of 70-90 psi.

c. Temperature – Increasing the temperature of the substrate above room temperature does not result in any advantage in the plating of thin TFE films.

d. Activity – Generally, for good even electrodeposition to take place, which is free from blisters and pits resulting from oxygen evolution, the work must be activated with an acid. The best results were obtained with uninhibited hydrochloric acid at a concentration of 10% by weight and an immersion time of 5 - 6 seconds.

2. Plating variables

a. Voltage – An increase in voltage generally causes an increase in the toughness of the wet deposit. In the plating of thin films, however, the increase is very slight.

b. Amperage – The effect of amperage is unimportant.

c. Anode current density – The rate of deposition is dependent on the current density employed. The current density also affects the character of the deposit. If the current density is too low, spotty deposits may result. If the current density is too high, oxygen evolution may take place, resulting in blisters and pits in the finished coating. However, a wide range of current densities may be employed without noticeably affecting the character of the deposits. Current densities in the range of 5 - 20 amperes per square foot are satisfactory.

d. Cathode current density – The effect of the cathode current density is unimportant.

e. Anode to cathode distance – As the anode to cathode distance increases a more even coating generally results. Distances of 1/2" to 6" are satisfactory.

f. Anode to cathode surface area ratio – The proper ratio is determined by trial and error as the normal rules of metallic plating do not apply.

g. Cathode configuration – The proper cathode configuration is determined by trial and error methods. This fact was also noted by other investigators (21).

h. Cell separations – Division of the plating bath into an anode compartment (containing the TFE dispersion) and a cathode compartment (containing an electrolyte), while practiced in the plating of heavy films, is not particularly advantageous for the plating of thin films. Enclosing the cathode within a porous glass fabric or sleeving,

8.

however, helps to contain the hydrogen bubbles formed in the cathode area. The use of a diaphragm results in a slight increase in the power requirements.

i. Rate of withdrawal — The rate of withdrawal determines to a degree the evenness of the deposit. A rate of 6 inches per minute is satisfactory.

3. Bath variables.

a. pH — The pH of the dispersion effects the quality of the deposit. Too low a pH results in thickness control difficulties caused by supplementary ionic reactions and too high a pH leads to passivity, causing spotty deposits. In the range of pH 4.0 - 7.0 excellent coatings are produced. The pH also effects the rate of deposition but this effect is unimportant when plating thin films.

b. Conductivity — In the range of conductivities employed in this study, little effect was noted by changes in the conductivity. Conductivity measurements are useful for quality control, however.

c. Concentration — The concentration of TFE solids in the bath determines the rate of deposition and the drainage properties. Below 25% solids, the rate of deposition decreases (unimportant in thin electrodeposits) and poorer drainage properties result. In the range of 25% - 35% solids, no pronounced effect is noted.

d. Temperature — In the plating of thin TFE coatings no advantage is noted by increasing the temperature of the bath. The upper temperature which could be employed is limited by the temperature at which the colloidal dispersion may be coagulated by heat.

e. Thixotropy — The thixotropic properties of the bath should be kept low to avoid pulling up too much "outer film" with the deposit.

f. Surface tension — The surface tension of the bath may range from 15 - 40 dynes per centimeter. Below 18 dynes per centimeter "crawling" may be experienced which could be a disadvantage in some instances.

g. Viscosity — The viscosity of the bath should be kept low to avoid pulling up too much "outer film" with the deposit.

h. Foam — To avoid defects caused by gas bubbles on the surface attaching themselves to the deposit as it is withdrawn, the foaming tendencies of the bath should be low and the foam should be unstable to heat so that once formed it is easily broken up.

i. Agitation — Rapid agitation of the bath is not necessary and could cause the bath to coagulate. Very slow agitation may be desirable to keep the pigments in better suspension.

j. Depth of immersion — As the current follows a parabolic path it is desirable to immerse the work at least two inches below the surface of the bath.

4. Bath constituents

a. Polytetrafluoroethylene (TFE) — The Teflon dispersion, of course, determines the basic properties of the coatings.

b. Wetting agents — The type and amount of wetting agent employed affects

9.

the "critical cracking thickness", bath stability, foam stability, and adhesion.

c. Pigments - The addition of pigments improves the appearance of the coatings by masking the yellow staining present in unpigmented coatings. Also, the addition of pigment tends to improve the CCT.

d. Electrolytes - The addition of electrolytes is of no advantage in the plating of thin films. Too much electrolyte in the bath will result in the formation of gas bubbles on the work piece.

e. Organic solvents - Organic solvents added to the bath in the form of emulsions is not advantageous in the plating of thin films.

f. Film-forming additives - The addition of film-forming additives such as Buna N rubber and polyisobutylene, results in an increase in the CCT of the bath. However, approximately 30% (based on the TFE weight) is required to produce crack-free films at .001 in. and considerable porosity results. In addition, these additives are not very stable in the desired pH range of 4.0 and most produce some coagulum on the surface. The plating rate of the additive may be sufficiently different from that of the TFE that maintenance of the proper additive to TFE ratio may be difficult.

g. Other additives - Addition of colloidal silica (alumina modified) results in a slightly harder coating. The CCT of the bath is lowered, however. The addition of fluorinated ethylene propylene (FEP) results in increased adhesion but slightly lower CCT.

5. Drying, sintering, and cooling.

a. Drying - The drying operation must take place soon after the coated panel is withdrawn from the bath. Normally, to prevent rust formation, simple fan drying is employed and is completely satisfactory. The panels may be further dried in a low temperature oven at 180°F. for 30 minutes.

b. Sintering - The time and temperature of sintering are important in the development of adhesion. This phenomenon is fully discussed in Heading "N".

c. Cooling - Normally the panels were allowed to air cool on removal from the oven. Quenching in cold water aids in the prevention of crazing and cracking with, however, some loss in adhesion.

II. EXPERIMENTAL

A. Materials.

The materials used during this study are tabulated in the Table XXXV . Any modification made prior to incorporating into the TFE baths are noted at the end of this Table or noted in the body of the report.

B. Explanation of Terms

The meaning of all terms or abbreviations used in this study are listed in Table XXXVI or explained in the body of this report.

C. Test Panels

The test panels used in this study were SAE 1010 or 1020 cold rolled steel. The dimensions of the panels were 1-1/4" wide x 3" long x 3/16" thick. To limit the size of the plating bath and to facilitate the calculation of thickness measurements by the weight/area method one face and all edges were first insulated with a sprayed TFE coating. The electrodeposit was applied to the uncoated face. The area of this face was 0.026 square feet.

D. Plating Set-up

The plating set-up employed was generally similar to that of any electrodeposition process except that the panel (or work) was connected to the positive side of a rectifier. The TFE dispersion was placed in an insulated oval container of approximately 350 cc. capacity. The panel to be coated was hung on a rod above the bath by a hook which was placed through a 1/4" hole near the top of the panel. The panel (or anode) was connected to the __positive__ terminal of a rectifier through a 0-1 amp ammeter, a relay and a 0-6 second timer (graduated in 0.1 second intervals). A 1/8" copper rod covered by a 3/16" O.D. glass sleeving was connected directly to the negative terminal of the rectifier and thus served as the cathode. The anode to cathode distance was 1-1/4" although this distance was not fixed. The source of DC current was a rectifier with a range of 0-150 volts and a capacity of 30 amperes.

E. Plating Bath Make-up

The methods and materials used to formulate the TFE plating bath were as follows unless otherwise noted.

1. Base Dispersion (Teflon 41BX) – The base dispersion used was Teflon 41BX, a product of the Plastics Department (formerly the Polychemicals Department) of the Du Pont Company. This dispersion is an aqueous dispersion of negatively charged TFE particles having an average diameter of 0.2 microns. The viscosity is 4 centipoises at room temperature and the pH is approximately 10. The dispersion does not contain wetting agents or other additives. It is stabilized for shipment by the addition of an immiscible oil. The oil is removed prior to use by placing the oil stabilized dispersion in a separatory funnel and drawing off the raw TFE dispersion after sufficient time has elapsed for the oil to rise to the top.

2. Reduction of the hydroxyl ion concentration – The pH (or OH^-conc.) of the Teflon 41BX dispersion is reduced from 10.0 to approximately 5.5 by slurrying with an

ion exchange resin (IRC-50). When the desired pH value is reached the TFE dispersion is filtered off. This ̣ ᷣᷣdure results in maximum flexibility of formulation as the desired pH range for ᷣplating thin TFE coatings is 4 - 7. This method for reducing the pH is preferred to ᷣf adding acids as it avoids the incorporation of large amounts of highly conductive ion ̣ such as chloride ions, etc. and limits the possible formation of water soluble salts in the coatings.

3. Reductio.ᶦ of the solids content - Normally, the solids content of the Teflon 41BX dispersion ̣ is reduced from 35.0% to 30.0% at this point by diluting with deionized water (conductivity = 5 - 7 micromhos/70°F.). Several times the solids content was reduced following the addition of all ingredients. This procedure will give essentially the same end result.

4. Compounding - Following the step of reduction in pH, a wetting agent was added to the unstable Teflon 41BX dispersion to stabilize it. The degree of stabilization achieved is dependent on the type and amount of wetting agent employed. Once stabilized, the pH was adjusted with ammonium hydroxide to the desired value and additives such as adhesion promotors, pigments, etc. were added. All formulations are based on weight percent and all ingredients added in amounts based on the solid TFE weight present in the dispersion except where noted. Unless otherwise noted, fluorinated ethylene propylene (FEP) and colloidal silica were modified according to the procedure shown in the appendix Table XXXV. Most other additives were added in the concentration and pH as supplied. Due to the simplified method of listing formulations, the total solids content (i.e. TFE plus all other non-volatile ingredients) will be slightly higher than the normal 30.0% TFE solids content. All wetting agents are assumed to be non-volatile matter for purposes of determining the solids content experimentally. The total wetting agent concentration may in some instances be slightly higher due to the inclusion of some wetting agent in the additive employed. However, the change is minor and by employing the data in the appendix Table XXXV, both the total solids content and total wetting agent concentrations may be determined, with the exception of pigment additives. The pigments used were one manufacturer's proprietary water dispersions and no data was supplied as to the amount and type of dispersing agents and/or wetting agents contained therein. As the amounts of pigment normally used was low and the amount of wetting agent added directly to the TFE dispersion fairly high, the true wetting agent content in the dispersion should be approximately the same as that reported or calculable. The slight excess would most likely be of a different variety of wetting agent.

F. Coating Procedure

The general procedure for coating the panels via electrodeposition methods was as follows unless otherwise noted.

1. Degreasing - The panels were degreased in trichlorethylene to remove any oils or greases present on the surface.

2. Sandblasting - The panels were sandblasted with #80 silica sand at 70-90 psi air pressure to produce the desired "satin" finish, as specified by the NRL report (3).

3. Activation - The sandblasted panels were immediately activated, where noted, by dipping in a 10% by weight solution of Hcl for 5 seconds.

4. Rinsing - Immediately following activation, the panels were rinsed thoroughly

in tap water, followed by a rinse in distilled water. The panel was then placed in the bath while still wet.

5. Plating – Immediately the timer button was pressed to apply current for the required time (pre-set). The voltage adjustment had previously been set to give the desired current density on the panel.

6. Withdrawing – Immediately the panel was withdrawn from the bath at a uniform rate of approximately 6 inches per minute. Hand withdrawal was used, although it would be expected that mechanical withdrawal would provide more even coatings.

7. Drying – Upon removal from the plating bath the panel was placed in front of a fan until the coating dried. Occasionally it was further dried by placing in a gravity type oven for 30 minutes at 180°F.

8. Sintering – Following fan or oven drying, the panel was placed in an oven maintained at 700°F. – 750°F. for the stated interval of time.

9. Cooling – Upon removal from the oven the panel was normally allowed to air cool, but occasionally it was quickly quenched in cold water. Unless otherwise noted, it can be assumed that the panel was air cooled.

G. Examination and Tests

Following the sintering and cooling steps, the electrodeposited coatings are examined visually and under a magnification of 24X or 27.6X.

1. Visual – The coatings are examined visually for evidence of bare areas, thin areas, poor pigment hiding, pigment flocculation, bare spots, blisters, pits, and other defects that effect the general appearance of the coating. Cracking and crazing are not generally visible to the naked eye.

2. Microscopic – The panels are examined under a microscope at a magnification of 24X or 27.6X to note, in addition to the above defects, the presence of cracking and crazing. The amount of cracking and crazing present in the coating is given by the terms "random", "light", "medium", "heavy", etc. An explanation of these terms and the method used to differentiate between cracking and crazing is given under Heading "N".

3. Adhesion – The adhesion of the electrodeposited coatings was originally measured by peeling built-up films. The film thickness of the electrodeposited coatings was increased to 9.0 mil or more by repeated applications of Du Pont Spray Finish #851-224 (High Build Green). This procedure resulted in some erroneous conclusions as to the adhesion of electrodeposited films as the adhesion generally increased with an increase in film thickness. The built-up method was used because at thicknesses of approximately 1.0 mil the TFE coating could not normally be stripped from the panel without tearing it, even when the adhesion was poor. This is because of the inherently low tensile strength of TFE coatings, both sprayed and electrodeposited. More meaningful results were obtained by attempting to strip the coating following each application of Du Pont Spray Finish #851-224. The adhesion was then evaluated at the minimum built-up film thickness at which the coating could be stripped without breakage or tearing. The procedure used was to place one end of the peeled back coating in the jaws of a clamp attached to a spring loaded testing device (a Hunter Force Indicator manufactured by the

Hunter Spring Company in Lansdale, Penna.) A force was applied by hand at a rate of approximately 12 inches per minute at an angle of approximately 90°. While the film was being peeled from the panel the maximum and minimum values obtained were noted from the circular dial and recorded. Although this method gave a numerical value, and use of the lower film thickness gave more meaningful results, the method still did not take into account the influence of the top coats applied or the effect of the additional sintering cycles involved.

A better method, although one which did not give a numerical rating, was to rate the resistance to peeling of the coating by the use of a knife or fingernail. This method was only applicable for film thicknesses of 1.0 mil or over, as difficulty was experienced in evaluating the resistance to peeling at lower thicknesses due to the low tensile strength of the coating. The adhesion is simply rated as "poor", "fair", or "good".

A better rating system using the above method was to assign code numbers based not only on the ease of stripping the panel with a knife or fingernail, but also on the ease of removing portions of the coating by gouging or ploughing. A description of this method of rating adhesion is described under the Heading "N".

In addition to the above tests, simulated use tests were conducted on selected specimens by the Naval Research Laboratory (NRL). This test employs a modified Bowden-Leben apparatus, a machine used to measure the frictional properties of materials and coatings. The test consists of repetitively cycling at room temperature a 0.5" steel ball for 100 reciprocating traverses at a speed of 0.1 cm./sec. under a load of 10,000 grams (approximately 160,000 psi) across the coated panel. Friction traces are produced during the test and photomicrographs made of the wear tracks following the conclusion of the test. Using the friction data and the photomicrographs of the wear tracks it is possible to rate the adhesion or durability of the electrodeposited coatings against sprayed TFE coatings on which considerable data has been developed.

Although the Bowden-Leben apparatus is the best method of evaluating the adhesion of TFE electrodeposited coatings, the apparatus was too expensive to warrant duplicate equipment and due to the time and expense involved in conducting these tests, only those coated panels which had previously shown evidence of improved adhesion, when rated by the other test methods, were submitted to NRL for test.

In general, evidence of improved adhesion obtained by the other test methods employed was substantiated by subsequent tests in the Bowden-Leben apparatus.

H.   TFE Dispersions Stabilized with Conventional Wetting Agents.

TFE dispersions for use in spraying or wire coating are stabilized with conventional wetting agents. The most popular wetting agents are alkyl aryl polyether alcohol and sodium lauryl sulfate. The CCT exhibited by these dispersions is approximately 0.0005". In spraying or dipping formulations the concentration of wetting agent is generally from 6.0% to 12.0% (based on the TFE weight). Previous plating experiments (22,23) employing baths stabilized with alkyl aryl polyether alcohol, sodium lauryl sulfate, or sodium octadecyl sulfate at concentrations of 1.5% - 6.0% (based on the TFE weight) were unsuccessful. The main failing of electrodeposited coatings produced from these baths was the lack of any increase in the CCT. In addition, these electrodeposited coatings exhibited poor pigment hiding and high foam stability.

14.

A series of electroplating baths were formulated with three types of sulfosuccinate wetting agents, namely, sodium diamyl sulfosuccinate, sodium dihexyl sulfosuccinate, and sodium dioctyl sulfosuccinate. These surfactants were added at a concentration of 3.0% (based on the TFE weight). Chrome oxide green pigment was added in concentrations up to 60.0% (based on the TFE weight). Electrodeposits produced from these baths exhibited no marked increase in the CCT, high foam stability, low bath stability with the higher pigment concentration baths, and a tendency toward pigment flocculation. The data is shown in Tables I, II, and III.

I. TFE Dispersions Stabilized with Conventional Wetting Agents and Perfluoroalcohols.

Bernett & Zisman(24,27)in their work with the addition of perfluoroalcohols to conventional wetting agents, report a marked lowering of surface tension by the use of select perfluoroalcohols. They found that by incorporating small amounts (10% of the total solutes) of insoluble omega hydroperfluorononyl alcohol to sodium lauryl sulfate and sodium dioctyl sulfosuccinate they could lower the surface tension of these solutions from 36.4 to 20.3 dynes per centimeter at 5°C. and 24.3 to 19.9 dynes per centimeter at 25°C., respectively. This substantial lowering of the surface tension they believed to occur through solubilization of the nearly insoluble omega hydroperfluorononyl alcohol in the micelles of water soluble conventional wetting agents. In attempting to incorporate perfluoroalcohols such as omega hydroperfluorononyl alcohol, omega hydroperfluoroheptyl alcohol, and perfluorooctanol into conventionally stabilized TFE dispersions, difficulty was experienced in solubilizing the nearly insoluble perfluoroalcohols. Although some reduction in the surface tension of the TFE baths was noted, most of the perfluoroalcohol was not dissolved. Electrodeposits produced from these baths exhibited a low CCT, poor pigment hiding, and no increase in adhesion.

More success was realized with the more soluble perfluorohexanol (designated ϕ C_6 alcohol). An electrodeposition bath was formulated with sodium dihexylsulfosuccinate and perfluorohexanol. The concentrations of the sodium dihexylsulfosuccinate and the perfluorohexanol were increased until the bath exhibited a surface tension of about 20 dynes per centimeter as evidenced by a visually estimated contact angle of 5°. The characteristics of the coatings produced from this bath were again a low CCT, poor bath stability, and poor adhesion. The data for this bath is shown in Table IV.

J. TFE Dispersions Stabilized with Highly Fluorinated Wetting Agents (other than Perfluorooctanoic Acid)

The highly fluorinated wetting agents show some unusual properties. Chemically, this class of surfactants consist of a long fluorochemical tail attached to a solubilizing organic group. Fisher and Gans (25) have shown that conventional, or nonfluorinated, wetting agents are unable to lower the surface tension of water to less than 26-27 dynes/cm. at 20°C. Some fluorinated wetting agents, on the other hand, will lower the surface tension of water down to 15 dynes/cm. at 25°C. Bernett and Zisman (26) showed that in order to completely wet a low energy solid such as Teflon, the surface tension of the water must be lowered below the critical surface tension of wetting of the solid Teflon, which they determined to be 18.5 dynes/cm. at 25°C. A number of highly fluorinated wetting agents were previously employed to stabilize TFE dispersions (23). These surfactants are products of the Minnesota Mining & Manufacturing Co. and designated FC-128, FC-95, and FC-98. Electrodeposits produced from TFE baths stabilized with these wetting agents at concentrations of 3.0% - 6.0% (based on the TFE weight) did not exhibit an increase in the CCT over

that exhibited by baths stabilized with conventional wetting agents. The electrodeposits also exhibited one or more of the following defects, namely: poor pigment hiding, poor bath stability, and poor adhesion.

1. Fluoroalkyl phosphate – This surfactant is a product of the Du Pont Company and sold under the trade name ZONYL-13. This surfactant was added to Teflon 41BX at a concentration of 3.0% by weight (based on the TFE weight) and the pH adjusted to 4.0 by the addition of ammonium hydroxide. The resulting dispersion was initially well stabilized but after several weeks the bath was completely coagulated. Electrodeposits produced from this bath before the addition of chrome oxide green pigment were very rough and agglomerated TFE particles were visible on the surface of the coating. The addition of the chrome oxide green pigment improved the appearance of the electrodeposits although some agglomerated TFE particles were still visible. The CCT of the bath, however, was not increased. The adhesion of the electrodeposited coatings to steel substrates was not increased. The data is shown in Table V.

K. TFE Dispersions Stabilized with Perfluorooctanoic Acid

Perfluorooctanoic acid, $C_7F_{15}COOH$ (or abbreviated ϕ octanoic) is a product of the Minnesota Mining & Manufacturing Company and designated FC-26. It is a white solid at room temperature and is only very slightly soluble in water (0.95% by weight). It has a melting point of 53°C. Bernett & Zisman (26) showed that before its solubility limit was reached, this surfactant could lower the surface tension of water to about 15 dynes/cm. at 25°C. and therefore would spread on a sintered TFE surface. Unlike conventional wetting agents or the other highly fluorinated wetting agents studied, this surfactant will increase the CCT of an unstabilized TFE dispersion.

The ϕ octanoic acid is added directly (with just enough water to make a slurry) to the Teflon 41BX dispersion after it has been treated with an ion exchange resin, either Rohm & Haas's IRC-50 or IR-120. The resulting changes in the pH and conductivity of the bath are illustrated by Figure 1. The procedure now followed in formulating electrodeposition baths from this surfactant is to treat with IRC-50, not IR-120.
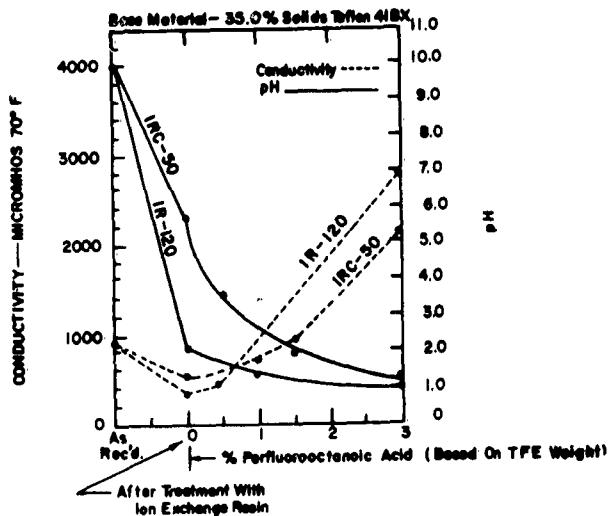
Figure 1. Effect of ion exchange resins and the ϕ octanoic acid concentration on the pH and the conductivity of TFE dispersions.

16.

Due to the high acidity of this wetting agent, TFE stabilized baths will produce deposits by simple immersion without benefit of an electromotive force (emf). The rate of deposition depends on the pH of the bath as illustrated by Figure 2. This phenomenon is caused by the high corrosiveness of the bath which releases ferrous ions (Fe^{++}) from the substrate, which in turn co-agulate the TFE particles. The mechanism is therefore similar to corrosion phenomena, or

$Fe^{o}-2e=Fe^{++}$ (oxidation at the anode)
$2H^{+}+2e=H_2$ (reduction at the cathode)
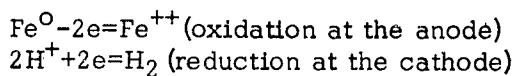
The surprising fact is that hydrogen gas is not formed on the coated panel for a brief period of time, although the anode and cathode must both be in the same general area. This time period, however, is normally sufficient to produce films up to 1.0 mil without the formation of blisters or pits. The very low surface tension of the bath may serve to remove any hydrogen gas formed, for a brief period of time.

Bath — 34.8% Solids Teflon 41BX
3.0% Perfluorooctanoic Acid (Based On TFE Weight)

Note: Shaded Area = Target Thickness Range

Figure 2. Effect of increasing pH on the ionic deposition rate.

Thus, in order to produce electrodeposits without the above supplementary reaction taking place it was necessary to raise the pH to 4.0 or above with ammonium hydroxide.

Electrodeposits produced from baths stabilized with 3.0% perfluorooctanoic acid (based on the TFE weight) at a pH of 4.0 were excellent in all respects and, in addition, allowed the coating in one operation of crack-free films up to a thickness of 1.5 mil. The adhesion of these coatings to steel substrates was very poor, however. Although the use of carbon black and chrome oxide green pigment had previously been investigated, the pigment concentration had been held to below 5.0% (based on the TFE weight). It was believed that possibly the addition of larger quantities of chrome oxide green pigment might serve to remove, by adsorption, excess ∅ octanoic acid present at the metal interface, thus increasing adhesion. This surfactant, being a low-energy solid, would, like Teflon, not tend to adhere to steel substrates. The data presented in Table VI, when compared to previous work (22,23) indicates that increased adhesion does result. The gain is slight, however. A slight problem with these baths was that as the pigment concentration increased, more rapid settling of the plating bath occurred and upon standing overnight considerable force was required to redisperse the pigment. The bath showed no sign of coagulation, however. As the pigment concentration was increased, the tendency toward pigment flocculation occurring during the drying cycle was increased. This resulted in uneven color distribution which, however, was only visible under a microscope. Continued use

of the bath increased the tendency toward pigment flocculation and in time the pigment appeared to be flocculated in the bath itself, although the bath stability with respect to TFE coagulation was still good. At a concentration of 20.0% chrome oxide green pigment these problems were minimized.

1. Investigation of the increase in CCT - The reason for the increase in the CCT of TFE baths resulting from the use of ∅ octanoic acid is not understood. It is, however, the only wetting agent studied which will increase the CCT significantly. Although the reason for this phenomenon is not understood, a number of factors which apparently are not the cause of this increase were determined from experimental data.

Although ∅ octanoic acid stabilized baths at concentrations of 4.0% or more exhibit very low surface tensions (as low as 15.0 dynes/cm.) and result in low pH readings (pH=1.5), increasing the surface tension and the pH by the addition of ammonium hydroxide does not cause a subsequent decrease in the CCT up to a surface tension of 28.0 dynes/cm. and a pH=7.0. TFE baths stabilized with wetting agents such as sodium dioctyl sulfosuccinate and fluorinated wetting agents such as FC-128 exhibit surface tensions below this value and yet these baths show no increase in the CCT.

As ∅ octanoic acid is added to unstabilized TFE, significant adsorption of the ∅ octanoic acid occurs onto the TFE particles. This fact is illustrated by Figure 3. By measuring the conductivity of the bath at various ∅ octanoic acid concentrations an estimate of the solubility limit in a bath composed of 30.0% solids Teflon 41BX and 2.0% carbon black (based on the TFE weight) was 5.0% based on the weight of the TFE. This concentration corresponds to 2.1% (based on the aqueous phase) or i.e. considerably above the solubility limit of 0.95%. The high adsorption of the ∅ octanoic acid onto the TFE particles is also illustrated by Figure 4. Here, the concen-
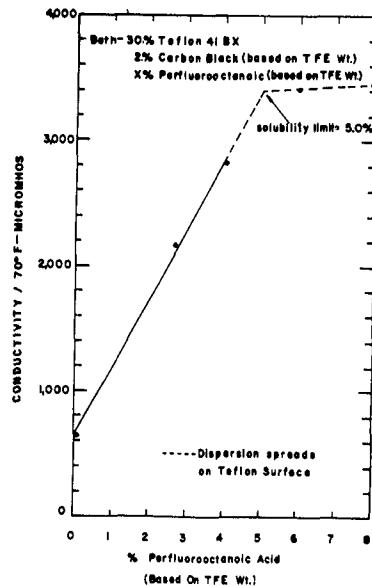
Figure 3. ∅ octanoic acid concentration vs. conductivity.
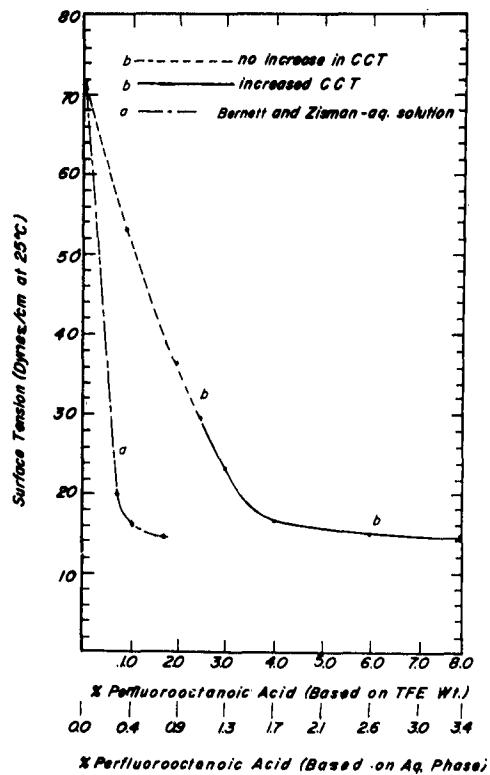
Figure 4. ∅ octanoic acid concentration vs. surface tension.

18.

tration of the ∅ octanoic acid (based on both the TFE weight and the aqueous phase) in a dispersion composed of 30.0% solids Teflon 41BX and 2.0% carbon black solids (based on the TFE weight) is plotted against the resulting surface tensions (curve b). Surface tension measurements were made with a Cassel Surface Tensiometer (28, 29).The surface tension vs. concentration curve for a solution of ∅ octanoic acid in distilled water is also plotted (curve a). The wide separation between the two curves illustrates that considerable adsorption of ∅ octanoic acid onto the TFE particles occurs. It is possible that the ∅ octanoic acid is adsorbed in sufficient quantities to act as a plasticizer and thus hold the resin particles together during the drying and sintering steps. The graph also shows that the increase in the CCT occurs at ∅ octanoic acid concentration of approximately 2.5% (based on the TFE weight). No further increase in the CCT occurs with increased ∅ octanoic acid concentrations.

L. Additives

Since TFE dispersions stabilized with ∅ octanoic acid exhibit poor adhesion to steel substrates when sintered at normal baking times and temperatures, various additives were incorporated in attempts to improve the adhesion of the electrodeposits without affecting the excellent plating characteristics of these baths.

The excellent adhesion of sprayed TFE coatings to steel and other substrates is obtained through the use of patented primers (30,31). These primers contain chromic acid or mixtures of chromic acid and phosphoric acid dissolved in the aqueous phase of the TFE dispersion. Previous attempts (22,23) to incorporate even small amounts of these materials in ∅ octanoic acid stabilized plating baths led to copious evolution of oxygen gas on the work piece, resulting in highly porous electrodeposits. This was expected due to the high conductivity of thes strong electrolytes.

Although is was possible to incorporate small amounts of conventional wetting agents into ∅ octanoic acid stabilized baths, the resulting electrodeposits showed no increase in adhesion over that exhibited by ∅ octanoic acid stabilized baths alone. Also, the addition of conventional wetting agents lowered the CCT.

Water soluble phenolic resins (BRL-1100), when incorporated into ∅ octanoic acid stabilized baths, did increase the adhesion of the electrodeposits. When added in the minimum concentration necessary to show improvement (20% based on the TFE weight), however, the TFE plating bath was coagulated after only several days' use.

Elastomeric type additives such as Buna N, Buna S, and polyisobutylene, while not lowering the CCT, did not improve the adhesion of the electrodeposits and tended to increase the porosity.

1. Colloidal alumina - "Baymal" is the registered trademark of the Du Pont synthetic colloidal alumina, a hydrated aluminum oxide (AlOOH). It is supplied as a free-flowing powder containing about 10% adsorbed acidic acid. When dispersed in water a positively charged colloidal dispersion is formed. Attempts to add small amounts of colloidal alumina (from a 5% aqueous dispersion) to ∅ octanoic acid stabilized TFE baths were unsuccessful. The TFE dispersion was either coagulated or a highly thixotropic gel was formed. While it was possible to add up to 26.0% colloidal alumina (based on the TFE weight) to unstabilized TFE (Teflon 41BX) dispersion, the adhesion to steel substrates (when checked on sprayed coatings by the built-up adhesion method) was low, ranging from 1.4 to 1.8 lbs/in. for various concentrations of colloidal alumina. Based on these experiments, therefore,

It would appear that even if a procedure were found to incorporate colloidal alumina into ∅ octanoic acid stabilized baths, the adhesion of the electrodeposits would not be improved.

2. Organic titanates – Du Pont produces a series of organic titanates under the trademark "Tyzor". The materials find use as adhesion promotors. Attempts to incorporate either Tyzor PB or Tyzor AA into ∅ octanoic acid stabilized TFE baths were unsuccessful due to the inability to form a stable emulsion within the TFE dispersion.

3. Fluorinated ethylene propylene (FEP) – Fluorinated ethylene propylene resin (FEP) is a copolymer of tetrafluoroethylene (TFE) and hexafluoropropylene (HEP) available from the Plastics Department of the Du Pont Company in the form of a 60.0% solids water dispersion (TE-9500). The average particle diameter is .015 microns and the pH, as supplied, is 9.0-11.0. This dispersion is believed to be stabilized with 6.0%, by weight, of alkyl aryl polyether alcohol (Triton X-100 – Rohm & Haas), based on the FEP weight. The dispersion exhibits a CCT of approximately 0.0003" (lower than conventionally stabilized TFE). When added to a ∅ octanoic acid stabilized bath it therefore tends to lower the CCT. FEP, unlike TFE, possesses fair adhesion to steel substrates. Although normally sintered at 600°F. – 650°F., the use of higher temperatures such as 700°F.-750°F. (the TFE sintering range) does not cause significant decomposition of the material.

Previous work (22,23) with the addition of various amounts of FEP dispersion to ∅ octanoic acid stabilized baths indicated that FEP increased the adhesion when added in amounts greater than 10% (based on the TFE weight). This fact was substantiated by subsequent tests with the Bowden-Leben apparatus conducted by NRL (32). This gain in adhesion, however, was not sufficient to equal or approach that obtainable with sprayed TFE finishes employing special primers. The tests also proved that in amounts up to 30.0% FEP (based on the TFE weight) the frictional properties were not affected. Although the CCT was lowered with increasing concentrations of FEP, up to 20.0% (based on the TFE weight) could be added without lowering the CCT below a usable value. The excellent stability of the ∅ octanoic acid stabilized TFE baths was not affected by the addition of the FEP but the drainage properties were slightly poorer. Before adding to the TFE dispersion, the solids content of the FEP dispersion was lowered to 30.0% and the pH reduced to approximately 4.0 by the addition of ∅ octanoic acid (see Appendix Table XXXV).

A compounded FEP dispersion spray finish recently became available from the Finishes Division of the Du Pont Company and is designated #856-200. This dispersion contains an unknown additive which makes it much less susceptible to flaking off when applied in heavy sprayed films than the TE-9500 dispersion. The CCT of the modified dispersion is no higher than that of the TE-9500 dispersion, however. The #856-200 dispersion was incorporated into the ∅ octanoic acid stabilized TFE baths at a concentration of 20.0% (based on the TFE weight). Chrome oxide green was also incorporated at a concentration of 20.0% (based on the TFE weight). Electrodeposits produced from this bath, although of good appearance, did not increase the adhesion more than the TE-9500 additive and no other advantage was found. This dispersion is also more expensive than the TE-9500 dispersion and no further work was done with it. The plating data is presented in Table VII. Further reference to FEP in later sections indicates the TE-9500 dispersion as modified according to procedure in the Appendix Table XXXV.

4. Colloidal silica (alumina modified)

Previous work (22) employing colloidal silica as an additive to TFE plating baths was not promising due to the fact that the stability of the colloidal silica modified TFE baths

was poor. The colloidal silica dispersion used in these experiments was Du Pont's "Ludox" HS. This dispersion did not have sufficient stability in the pH range employed for the electrodeposition of TFE coatings, i.e. pH = 4.0 - 6.0. Recently a new colloidal silica dispersion was introduced with had the necessary stability in the pH range involved. This material is designated "Ludox" AM and is also manufactured by Du Pont. The surface of the colloidal silica particlesin this dispersion have been modified by substituting aluminum atoms for part of the silicon atoms. As the aluminum is trivalent and the silicon tetravalent the unbalanced electrical charge that results is corrected by adsorption of a hydroxyl ion from the aqueous phase. Since this hydroxyl ion is negatively charged, the particle acquires a much stronger negative charge than exists on the unmodified silica particle. The net result is a much more stable dispersion in the pH range of 4.0 - 7.0. "Ludox" AM is supplied as a 30.0% solids anionic aqueous dispersion at a pH = 9.0. The average particle diameter is 0.015 microns.

Alumina modified colloidal silica when added in a concentration of 10% (based on the TFE weight) to a TFE dispersion composed of 30.0% Teflon 41BX plus 3.0% ∅ octanoic acid (based on the TFE weight) resulted in a very stable dispersion. Before adding to the TFE dispersion, the pH of the alumina modified colloidal silica was reduced to 4.0 by adding ∅ octanoic acid. (See Appendix Table XXXV for procedure.) Electrodeposits produced from this bath were smooth and even (except where too thin or not acid etched) although they were stained brownish-yellow, which is usual when no pigment is present to mask the discoloration. The addition of the alumina modified colloidal silica did lower the CCT of the basic bath. The adhesion, when measured by the built-up method, however, was apparently increased, although reproducability was poor. In several instances the adhesion of the silica modified TFE electrodeposits to the steel substrate exceeded that between the electrodeposit and the applied topcoat. This was evidenced by the fact that portions of the electrodeposited films remained on the substrate following the peel test. The data is shown in Table VIII.

M. Pre-treatment of the Substrate

In addition to incorporating additives in the bath itself, attempts to improve the adhesion of ∅ octanoic acid stabilized TFE baths were made by treating the substrate with various materials. Treating the substrate with dilute solutions of chromic acid and phosphoric acid mixtures prior to placing in the electroplating bath resulted in the same defect noted when these materials were added to the bath itself, i.e. copious oxygen evolution on the work piece.

Pre-treating the substrate with dilute solutions of water dilutable phenolic resins (BRL-1100) or water soluble urea and melamine formaldehyde resins (Accobond 3810) prior to electroplating did not result in increased adhesion.

Pre-treating the substrate with dilute solutions of FEP resin prior to electrodeposition exhibited some increase in adhesion. However, the electrodeposited panels exhibited many more defects such as blisters and pits than when the FEP was incorporate into the bath itself. Incorporating FEP directly into the bath itself is the preferred method.

1. Hinac* Process - The "Hinac" I-X process is a method of producing a thin (.0000004" - .000016") corrosion resistant chromium-containing coating on steel substrates. It is a patented process of the Pennsalt Chemical Corporation. It consists of applying a chromic acid solution containing a reducing agent and then baking the coating briefly to insolubilize it.

A number of experiments were conducted in which the steel panels were given a Hinac coating before the TFE was applied over it by electrodeposition methods or by spraying. The procedure for applying the Hinac was to sandblast the panel, dip briefly at room temperature or higher in the Hinac solution and then bake for ten minutes at 400°F. to insolubilize the coating. A number of different procedures were employed as shown by the data given in Table IX. The adhesion was not significantly improved with the one exception wherein a thin TFE/FEP dispersion (Formula A) was applied by spray methods. This formula, due to its very high percentage of FEP, cracks at a value below the required thickness range 0.0006" – 0.001". Electrodeposits applied over the Hinac show no evidence of increased adhesion.

2. Chemical etching – A recent patent issued to Cahne (33) describes a method of treating a metal base in such a manner that a subsequently applied coating of unmodified TFE will adhere to it. The theory behind this method of obtaining adhesion is based on the idea that by choosing the correct etchant and etching conditions cavities can be produced in the metal that will have a throttled entrance. If the cavities are of sufficient size to contain the TFE particles, the particles would be anchored within the cavities and considerable force would be required to strip the coating. This invention is mainly concerned with increasing the adhesion of TFE to aluminum substrates. Although etchant formulations and conditions are given in the body of the patent for increasing the adhesion to steel substrates, these methods do not appear in the claims section. The patent does not give any specific adhesion data except to mention cross hatch test procedure.

Employing the approximate conditions given in this patent, panels were etched and then TFE coatings applied by spraying or electrodeposition methods. Several difficulties were experienced. First, the nitric acid etchant removed considerable metal and the dimensions of the panels were changed. Secondly, although etched at the same time, the panels did not exhibit the same evenness of etch. Attempts to improve the evenness of etch by the addition of 0.1% of a fluorocarbon wetting agent (FC-95) were not successful. In fact, panels etched in this bath were even more uneven. Results obtained by treating the etched panels with a number of different formulations and coating methods are shown in Table X. Some instances of increased adhesion are indicated, although not when the panels were coated by electrodeposition methods. Difficulties were encountered when electrodepositing over nitric acid etched panels. This was due to the fact that nitric acid passivates steel. Even a long etch or "activation" in 10% HCL at high temperatures did not appreciably reactivate the steel. It was necessary to re-blast the etched panel in order to "reactivate" it.

The results obtained on etched panels with ∅ octanoic acid stabilized TFE baths and ∅ octanoic acid stabilized TFE baths containing small amounts of FEP are shown in Table XI. Although the data indicates improved adhesion when FEP additive is included in the TFE bath, the gain is not significantly improved over that exhibited by the same bath on sandblasted panels. Also, the surface finish of the coatings is much rougher and generally unsatisfactory in appearance.

3. Organic titanates – Organic titanates such as Tyzor PB and Tyzor AA were used to pre-treat the substrate prior to electrodepositing from ∅ octanoic acid stabilized TFE baths. No improvement in adhesion of the coatings was indicated and the general appearance was only "fair".

N. Extended Sintering Times or Temperatures.

During the experiments with alumina modified colloidal silica additive, the sintering times and temperatures were increased above those normally employed and a noticeable increase in adhesion appeared to result when rated by attempts to strip the panels with a knife or a fingernail. The differences in adhesion were simply rated "Poor", "Fair", and "Good". Although admittedly crude, the possible influence of the applied topcoats in the built-up method was eliminated in this method and the apparent difference in adhesion between a "Poor" and "Fair" rating appeared, to the author, to be quite large.

The adhesion of electrodeposited coatings produced from baths containing 10% (based on the TFE weight) of colloidal silica appeared to be increased when the sintering times and/or temperatures were in excess of those normally employed, (see Table XII and Table XV). This was not noticeable when the colloidal silica content was increased to 20% (based on the TFE weight), where apparently normal sintering times and temperatures were sufficient to develop "Good" adhesion, (see Table XII and Table XIV). However, this might be explained by the fact that the coatings were generally thinner and were more highly cracked than when the colloidal silica content was only 10%. Although highly cracked when no pigment was present (it is difficult to note cracks in clear coatings), the addition of 20.0% (based on the TFE weight) of chrome oxide green pigment to a bath with only 10% colloidal silica improves the CCT enough to allow mainly crack-free coatings to be produced. This is not so when the bath contains 20% colloidal silica (based on the TFE weight). These baths are still highly cracked. It was noted that in order to make a good evaluation of the adhesive properties of the coatings, the film thickness had to be at least 0.001".

When electrodeposited coatings produced from baths containing no colloidal silica additive were given extended sintering times, surprisingly, the same effect was noted as illustrated by the data in Table XVI.

Several electrodeposited coatings in this series were submitted to NRL for testing in the modified Bowden-Leben apparatus. The results of these tests is tabulated in Table XVII, the panel numbers corresponding to those in Tables XII - XVI. The photomicrographs taken of the wear tracks following the conclusion of the test and on which the comments in Table XVII are based, are not reproduced here. From these tests it was concluded that:

1. Increased sintering times and/or temperatures above normal are beneficial in increasing the resistance of the coatings to lifting or removal under high loads.

2. The coefficient of friction of electrodeposited TFE coatings is not significantly increased by incorporating medium amounts of colloidal silica and/or chrome oxide green pigment.

3. Where "extrusion" of the coating was noted, this was the result of too high a film thickness. As previously mentioned, these panels were purposely produced at thicknesses of 1.0 mil or more so as to better rate them for adhesion by the method just described.

4. The satisfactory result obtained in the one case (Panel #92) where no colloidal silica was present in the coating, indicates the possibility that the adhesion of all electrodeposited coatings may be increased by extending the sintering times and temperatures.

As the preliminary experiments with increased sintering times and/or temperatures were encouraging, further work was conducted along these lines. Before the new experiments were undertaken, the temperature indicator attached to the oven used throughout this work was tested for accuracy by a company specializing in this business. The indicator was found to be reading 40°F. - 50°F. higher than the true oven temperature. Thus the true oven temperatures are 40°F. - 50°F. below those values just reported. In most instances this still means that the previous panels were sintered for periods longer than normal. The oven temperature indicator (the thermocouple for which is located outside the oven work zone) was then adjusted. In addition, a newly rebuilt Leeds & Northrup "Micromax" indicator was attached to a thermocouple which was placed in the oven zone at a point just above the area where the coated panels were introduced. The two indicators did not agree and varied as follows:

Permanent Oven Temperature Indicator (thermocouple located outside the oven work zone)	Leeds & Northrup "Micromax" Temperature Indicator (thermocouple located above the work in the oven work zone)
603°F.	625°F.
607°F.	635°F.
612°F.	645°F.
640°F.	670°F.
700°F	720°F.
750°F.	760°F.
800°F	815°F
850°F.	872°F.
900°F	910°F.
1000°F.	990°F.

It was found that when a TFE coated panel was introduced into the oven at the following indicated temperatures it did not sinter even with sintering times of 40 minutes or more:

Oven Indicator	Leeds & Northrup "Micromax"
603°F.	625°F.
607°F.	635°F.

The TFE Films became sintered when the indicators showed:

Oven Indicator	Leeds & Northrup "Micromax"
612°F.	645°F.

If, as believed, TFE films will sinter once they attain a temperature of 621°F., then the true oven temperature must be approximately half way between the oven indicator reading and the Leeds & Northrup "Micromax" indicator reading. For simplicity, the oven temperature readings are recorded from the oven indicator and all surface temperature readings are recorded from a thermocouple attached to a Leeds & Northrup "Micromax" indicator. The oven used in this and previous work is manufactured by The Modern Laboratory Equipment Co., New York, N. Y. to the following specifications:

Temperature Range - $0-1200^{\circ}$F.
K.W. - 6.8

Heater Switches - 3 ranges (Hi-Med-Low)
Oven Chamber - 19" x 19" x 19" Stainless Steel
Insulation - 6" glass wool
Fresh Air Inlet Vent - 2" diameter
Exhaust Vents - 2-2" diameter
Recirculating Blower - 6" diameter (1/2 H.P. Motor)
Recirculating Damper - 0 to Full Range Circulation
Heater Cycling - $\pm$ 2°F.

During the following experiments the oven variables were maintained as follows:

Fresh Air Inlet Vent - 1/4 open
Recirculating Damper - 3/4 open.
Heater Switches - Hi
Exhaust Vents - slightly open

With the oven variables maintained constant a number of panels were electrodeposited with TFE to a thickness of approximately 0.001" from a bath composed of:

30.0% Teflon 41BX
3.0% ϕ octanoic acid (based on the TFE weight)
10.0% alumina modified colloidal silica solids (based on the TFE wt.)
20.0% chrome oxide green pigment solids (based on the TFE weight)

The resulting electrodeposits were sintered at normal times and temperatures and at extended (or abnormal) times and temperatures. Examples of the wide difference between these sintering schedules when sintering a 0.001" TFE coating on a 3/16" thick steel panel are shown below:

Oven Sintering Temperature	Normal Sintering Time (Minutes)	Extended Sintering Time (Minutes)
700°F.	10	120
750°F.	7	45

Also sintering oven temperatures of 800°F.-1000°F. were employed, temperatures which are not normally employed in sintering TFE. The data for this series of experiments is tabulated in Table XVIII.

When evaluating the adhesion of the sintered TFE coatings, it was again evident that increases in sintering times and/or temperatures over those normally employed increased the adhesion of the coatings to steel substrates when rated as to the relative ease of stripping a continuous film from the panel with a knife or thumbnail. A more accurate method of rating the adhesion was developed which took into account the effect on durability of over-sintering which the previous rating system of "poor", "fair", or "good" did not. It was noticed that as the sintering time at a particular temperature was increased, the electrodeposited films, which were at first readily strippable, became increasingly more difficult to strip from the panel. A point was soon reached where, although the films were not easily stripped by peeling, they were more readily gouged and more easily removed by scraping. This was due to the increased softness of the films, i.e. they became doughy, a definite indication of over-sintering. Obviously some point between

these two extremes would be best from the standpoint of durability in use. In rating the adhesion to show undesirable over-sintering, a rating of "#5" was considered the optimum as at this point the coatings exhibited both high peel resistance and high gouge or scrape resistance. Numbered ratings moving away from the mean of "#5" (i.e. towards "#1" or "#10") indicate increasingly lowered adhesion or durability. A list of the various factors taken into account in arriving at an individual adhesion rating is shown below:

Adhesion Rating	Ease of Starting a Strip	Ease of Peeling a Strip	Film Strength	Film Hardness	Ease of Gouging or Removing the Coating with Thumbnail
#1	Easy	Easy	Excellent	Hard	Difficult
#2	(increasing	(increasing	V.Good	Hard	Difficult
#3	difficulty)	difficulty)	Good	Hard	Difficult
#4	↓	↓	Fair	Hard	Difficult
#5	Difficult	Difficult	Fair	Hard	Difficult
#6	V.Difficult	V.Difficult	Fair	Softer	Less Difficult
#7	Impossible	Impossible	Poor	(increas-	(decreasing
#8	Impossible	Impossible	Poor	ing soft-	difficulty)
#9	Impossible	Impossible	Poor	↓ ness)	↓
#10	Impossible	Impossible	Poor	Soft	Easy

In addition to the sintering schedules, Table XVIII also shows the peak surface temperature attained by the coated panels while in the oven. The value was obtained by attaching a thermocouple to the panel before introducing it into the oven and thereafter recording the surface temperature of the panel at intervals as it was heated up and cooled. This data is listed in Table XIX. In each column the temperature which is underlined indicates that point at which the panel was removed from the oven. From that point the panels were cooling. All panels were cooled in air, not water quenched, except where noted.

Using the data in Table XVIII it was possible to construct a graph to illustrate the most desirable sintering time at a particular temperature to obtain high peel resistance and high gouge resistance. The following graph was constructed as follows: first, the sintering schedule of all panels was plotted as <u>Oven Temperature</u> vs. <u>Time</u> (in the oven). The points on the graph identifying the individual panels are not designated on the graph but can easily be found by referring to Table XVIII. Instead of employing the time the panel was in the oven, the time the panel surface was above 621°F. could have been used and would have given essentially the same result. At or above a temperature of 850°F. the surface of the coated panels never attained the oven temperature. Each point on the graph (representing the appropriate panel) was then assigned its "Adhesion Rating". Assuming the ideal sintering range to be only slightly removed from the mean of "5", or i.e. between adhesion ratings of "4" and "6" and connecting all points rated "4" and all points rated "6" (or their estimated location where no definite points are established) two curves result. The area lying within these curves thus represents the ideal or best sintering cycles. Areas below these curves are considered to be "under-sintered" from the standpoint of maximum adhesion development (not physical properties of the film). Areas above these curves are considered to be "over-sintered" as indicated by increasing polymer degradation taking place (i.e. increasing softness in the films. The mark (→) represents the approximate time it takes for the coated panel to reach 621°F. or, i.e. to reach the minimum sintering temperature. The normal sintering time (700°F. - 750°F.) would be slightly to the right of this mark.

26.
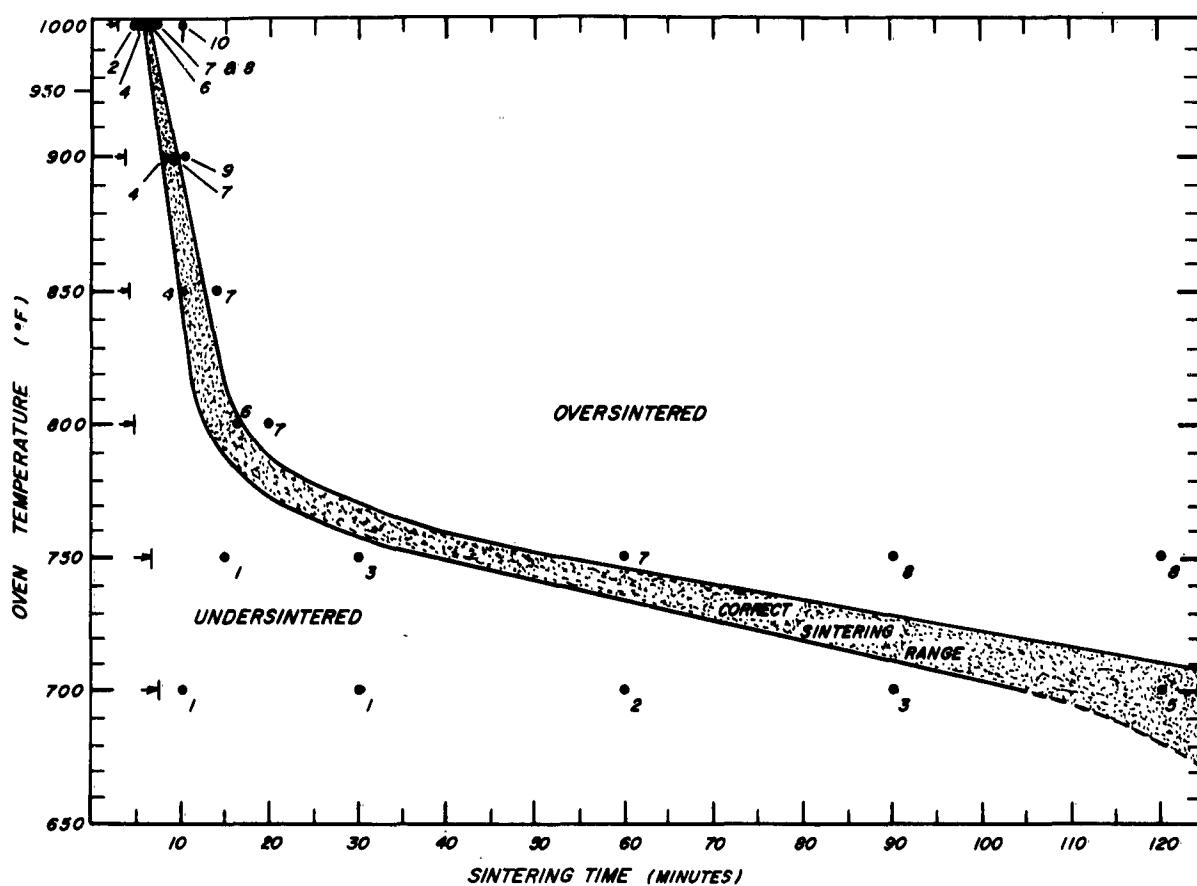
This graph clearly shows that in the sintering range of 700°F. - 750°F. greatly extended sintering times are necessary to obtain an increase in the adhesion. From an analysis of the data in Tables XVIII and XIX it appears that: (1) In developing adhesion the time the panel is at a particular temperature or increasing temperature is the important factor. The peak temperature reached is not important as even at low temperature (720°F.) adhesion is developed in time; and (2) Immediately quenching a panel in water when the surface temperature of the panel is in the range of 800°F. can result in lowered adhesion.

Further experiments were conducted for the purpose of (1) definitely determining whether or not alumina modified colloidal silica was required in the formulation to produce increased adhesion or durability when extended sintering times and/or temperatures were employed and (2) producing thinner, crack-free, well adhered coatings for further coefficient of friction, or durability tests by the Naval Research Laboratory (NRL). Electrodeposited coatings were produced from baths stabilized with ∅ octanoic acid and containing no colloidal silica additive, only increasing amounts of carbon black pigment. These coatings were sintered at extended times and temperatures and the same effect of increased adhesion with increased sintering times occurred. In later work with additional formulation containing no colloidal silica the same effect was again noted. Thus, it was definitely established that colloidal silica need not be in the formulation to increase adhesion by the use of extended sintering times. The data supporting this conclusion is found in Tables XX and XXI. These panels were sintered at an oven temperature of 800°F. and although, as shown, increased adhesion can be developed, if the correct time is employed, at temperatures up to 1000°F., later work was conducted at 700°F. - 750°F. For a commercial process this sintering range is more realistic and the time periods need not be so closely controlled.

27.

Continued investigation of other plating formulations revealed that many formulations produce coatings which tend to "craze" when sintered for extended periods. "Crazing" had been experienced before but never to this degree. Generally, it was found that the longer the sintering time or the higher the sintering temperature employed, the more severe the crazing. The phenomena of crazing is described as "minute cracks present on the surface of the coating". As they exist close to the surface and do not extend into the coat- ing, they are not to be considered as severe a defect as "cracks" which extend to the base metal. Nevertheless, since they are minute cracks, they must be considered as defects even if they do not extend to the base metal as they represent weak spots in the coating. When viewed at a magnification of 24X the crazing reported did not extend to the base metal. If viewed at 100X, several craze marks did extend to the base metal, although the width of the crack was much narrower than found in a normal crack. A magnification of 24X was used in differentiating between "cracking" and "crazing". The terms used to show the difference in the amount of cracking and crazing found on the panels are listed below:

No (No) - 0% - 1% of coated area covered
Random (Rdm) - 1% -5% of coated area covered
Light (Lt) - 5%-20% of coated area covered
Medium (Med) - 20%-40% of coated area covered
Heavy (Hvy) - 40%-80% of coated area covered
Very Heavy (v Hvy) - 80%-100% of coated area covered

Since crazing is no more than light cracking it was sometimes difficult to differentiate between the two. However, as the type of cracking usually experienced with electrodeposited coatings is one of several wide deep splits within a certain area, and that of crazing of many hairline cracks within the same general area, the difference in appearance is sufficient to rate them individually.

The data resulting from all further experiments with electrodeposited TFE coatings is listed in Tables XXII - XXVII and Tables XXX- XXXIII. The coatings were generally produced in two thickness ranges as required by NRL for their subsequent tests in the Bowden-Leben apparatus. Other thicknesses were produced during studies of the minimum thickness required for good pigment hiding, crazing, and other phenomena. Various formulations were employed but all were based on 30.0% Teflon 41BX plus 3.0% ø octanoic acid. To this basic formula was added carbon black pigment, lampblack pigment, chrome oxide green pigment, FEP alumina modified colloidal silica individually or in combination. The concentration of the additive was varied in most cases. The sintering temperatures ranged from 700°F. - 750°F. and the sintering times varied considerably.

Rather than discuss each series of experiments individually, the various factors influencing the quality of the electrodeposited coatings and the general merits of each additive are discussed below. For purposes of this discussion, the formulation

30.0% Teflon 41BX
3.0% ø octanoic acid (based on the TFE weight)

is described as the "basic formula".

General Appearance (naked eye) - To the naked eye all the panels appeared good or excellent (except where the coating thickness was below 0.4 mil.

Adhesion
    a. The adhesion of coatings produced from all formulations was increased when extended rather than normal sintering cycles were employed. Only spot checks were made

28.

so as to save the panels for test and the data is not reported. The best adhesion, however, was not as high as that exhibited by a sprayed TFE coating (i.e. green primer plus clear.)

b. Quenching the panel in cold water resulted in some loss in adhesion.

Hiding
a. At least 20% chrome oxide green pigment (based on the TFE weight) must be added to the basic formula to produce full pigment coverage in the middle areas of the panel at an average thickness of 0.8 mil.

b. At least 2.0% carbon black pigment (based on the TFE weight) must be added to the basic formula to produce full pigment coverage in the middle areas at an average thickness of 0.8 mil.

c. Higher current density improves coverage in the middle areas at an average thickness of 0.8 mil, but control of coating thickness is more difficult.

Roughness
a. Chrome oxide green pigmented formulations produce smoother coatings than either carbon black or lampblack pigmented coatings but the difference is slight.

b. Alumina modified colloidal silica additive in the formulation results in smoother coatings, but again the difference is slight.

Pigment Flocculation
a. Carbon black or lampblack pigmented formulations show little tendency towards pigment flocculation.

b. Chrome oxide green pigmented formulations show an increased tendency towards pigment flocculation especially as the bath ages.

c. The tendency towards pigment flocculation of chrome oxide green pigmented formulations increases with increased pigment content.

Bath Stability
a. All formulations show excellent stability towards coagulation.

b. Black pigmented formulations show far greater resistance to rapid settling than chrome oxide green pigmented formulations

c. Increasing the chrome oxide green pigment content increases the tendency towards rapid settling.

d. Chrome oxide green pigmented formulations do not settle sufficiently "hard" to prevent redispersion but considerable force is required to redisperse after only overnight settling.

Cracking
a. Increasing the sintering time over that normally used increases the tendency towards cracks.

b. The addition of carbon black, lampblack, and chrome oxide green pigment to the basic formula generally increases the CCT and therefore decreased the tendency toward crack formation.

c. The addition of additives such as FEP and alumina modified colloidal silica to the basic formula lowers the CCT and thus increases the tendency toward crack formation.

Crazing
a. Increasing the sintering time over that normally used increases the tendency toward crazing.

b. Quenching the panels in cold water decreases (in some instances markedly) the tendency toward crazing.

c. The addition of additives such as FEP and alumina modified colloidal silica increases the tendency toward crazing.

d. Cooling the coated panels to below 620°F. by shutting off the oven and allowing the panel to cool with the door closed in some instances lessened the tendency toward crazing. In other instances it did not.

Data resulting from the production of <u>sprayed</u> panels are listed in Tables XXVIII and XIX. The spray panels were produced in thickness ranges of 0.8 mil and 1.2 mil and in two colors, green and black. These coatings were submitted to the Naval Research Laboratory along with selected electrodeposited coatings of the same general thicknesses so as to compare the relative durability or adhesion of each type. Although the heavier thickness (1.2 mil) is not recommended for dry lubricant service, this thickness could show up any differences in relative durability when compared to electrodeposited coatings of the same thickness. Only a few panels were produced with green primer alone in the range of 0.8 mil due to the formation of cracks in the coating. A two-coat system (green primer plus clear) was substituted. The coating and sintering of the sprayed panels was done according to the methods recommended in NAVWEPS-OD 10362 ("Process of Applying Thin Films of Poly-tetrafluoroethylene Resins on Steel Surfaces").

A number of electrodeposited and sprayed TFE coatings submitted to NRL were evaluated by them in the modified Bowden-Leben apparatus. Although time did not permit a thorough evaluation, a sufficient number of coated panels were tested to indicate the effect of extended sintering times and/or FEP and alumina modified colloidal silica additives on adhesion. Also, a comparison of adhesion was obtained between electrodeposited and sprayed TFE coatings. Only chrome oxide green pigmented electrodeposited coatings were tested but it would be expected that black pigmented electrodeposited coatings would perform as well. The data sheet tabulating the results appears in the Appendix, Table XXXIV and is supplied through the courtesy of Mr. V. G. Fitzsimmons of NRL. Photomicrographs of the appearance of each wear track following the tests were also submitted but are not reproduced in this report. The code numbers appearing in Table XXXIV correspond to selected coated panels found in Tables XV-XIX. The measured coating thickness (taken in the middle of the panel) and noted in Table XXXIV was measured by NRL with a G.E. thickness gauge (Type B). These measurements agree closely with the average (or, as noted, estimate) coating thickness calculated by the author from area/weight measurements. Following completion of the tests, relative ratings as to the adhesion or durability was assigned by NRL to the three different electroplating formulations and the two spraying systems. They rate electrodeposited coatings produced from a bath containing FEP additive just below the best sprayed coatings. Electrodeposited coatings produced from the two other bath formulations were rated below the second best sprayed coatings. To clarify and make this data more meaningful, selected quotations from the test report submitted by Mr. V. G. Fitzsimmons, Head, Friction & Wear Section, Surface Chemistry Branch, NRL, along with the tabulated results and photomicrographs follows:

"4. As requested by BuWeps, Code RRMA-36, Code 6104 of this Laboratory has screened the various specimens of Teflon coatings supplied by General Plastics Corp. Comparative tests were performed in a Bowden-Leben Stick Slip Apparatus with both electrodeposited Teflon and with spray-coated Teflon. These tests had the primary objective of comparing the coatings for adhesion under extremely high frictional loading.

5. Past experience at this Laboratory with the Bowden-Leben Apparatus has established that repetitive cycling for 100 reciprocating traverses with a 0.5" steel ball at 10,000 gram

load (approx. 160,000 psi) will detect defective Teflon lubricant dry films having poor adhesion and/or cohesion. Additional information obtained during such experiments shows the effect of excessive film thickness and cold flow as well as the intermittent effect of coating ruptures which may become healed.

6. In every case where the specimens had a coating in excess of 0.8 mils there was the dynamic effect of hysteresis or frictional energy required to produce yield or displacement of the bulk coating. When such effects occurred friction was variable and coating durability was decreased.

7. The tabulated results, and the photomicrographs which have previously been delivered to General Plastics Corp., together form the basis for the NRL Rating on the right margin of the attached Table. However, it should be emphasized that only with the thinner coatings is such a rating possible.

8. It is obvious that, with the exception of the group of specimens which were produced by electrodeposition of TFE and chrome oxide, all coatings examined had adequate adhesion and resistance to mechanical penetration. The major visible differences between any specimens of a single type or even between different types occurred because of:

      a. Coating thickness and the properties associated with the internal structural behavior or the coatings.

      b. The sprayed coatings demonstrated a lack of adhesion between the primer and the top coat when the combined films were 1.0 mils or greater in thickness.

9. Within the limits of the experiments it can be concluded that the electrodeposited Teflon has adhesion to ferrous substrates which is quite comparable to the adhesion of sprayed Teflon coatings. Where the conditions of friction differ from those of the Bowden-Leben Apparatus, it would be unwise to extrapolate this information to predict behavior in another environment without appropriate experimental evidence."

      The author discussed the test data and viewed the photomicrographs at length with Mr. Fitzsimmons and concurs with NRL's rating of the TFE coatings.

DISCUSSION OF RESULTS

A. General

From the previous discussion, it is apparent that coatings produced from TFE baths stabilized with ∅ octanoic acid and incorporating 20% FEP (based on the TFE wt.) possesses adhesion approaching the best obtainable with <u>sprayed</u> TFE coating systems. Coatings produced from the other baths examined were not as well adhered. However, from an overall plating standpoint, these baths are more desirable. A comparison of the three baths is tabulated below. In this tabulation the bath formulas are as follows:

Bath A –   30.0% Teflon 41BX
              3.0% ∅ octanoic acid (based on the TFE weight)
             20.0% chrome oxide green pigment (based on the TFE weight)
             20.0% FEP solids (based on the TFE weight)

Bath B –   30.0% Teflon 41BX
              3.0% ∅ octanoic acid (based on the TFE weight)
             10.0% colloidal silica solids (based on the TFE weight)
             10.0% FEP solids (based on the TFE weight)
             20.0% chrome oxide green pigment (based on the TFE weight)

Bath C –   30.0% Teflon 41BX
              3.0% ∅ octanoic acid (based on the TFE weight)
             20.0% chrome oxide green pigment (based on the TFE weight)

The pH of the three baths was approximately 4.0

The slight differences observed with each bath, with respect to the various properties that determine the overall plating characteristics of the baths and subsequent quality of the electrodeposits, are rated as follows:

#3 – good
#2 – better
#1 – best

	Bath A	Bath B	Bath C
Adhesion (based on NRL tests)	1	2	3
CCT	3	2	1
Throwing Power	3	3	3
Foam Stability (Poor stability is desirable)	3	2	1
Bath Stability (Resistance to coagulation)	3	2	1
Hiding Power	3	2	1
Pigment Settling	3	1	2
Drainage Properties	3	2	1
Pigment Floculation	3	1	2

As previously illustrated, although Bath A is best for adhesive properties, it is the least desirable from the standpoint of overall plating characteristics. Bath C which exhibits the best plating characteristics, exhibits the poorest adhesion. Bath B is intermediate between the two. Although satisfactory electrodeposited coatings are produced from all baths, for applications not requiring the ultimate in adhesion, plating baths formulated without FEP would be more desirable.

Although the data in Table XXXIV does not indicate it, an examination of the photomicrographs of the wear tracks exhibited by panels #266 and #270 (coatings produced from Bath A) shows a slight superiority in panel #266 over panel #270. Panel #266 was sintered for a longer period of time than panel #270. Before any conclusion as to the advantage of extended sintering times can be drawn, it must be remembered that the best panel (#266) was also the panel with the thinnest coating. Since the latest NRL tests (and a previous investigation into the use of TFE as a dry lubricant film by NRL (3)) shows that with a particular formulation, the most singularly important factor affecting durability is film thickness, the fact that panel #266 was thinner than #270 would explain its slight superiority. However, when comparing the performance of thicker films, it can be reasonably concluded that increased sintering times over those normally employed results in improved adhesion. This is true because, although the latest coatings were extruded, they withstood the 100 cycles under heavy load (10,000 grams) without being severely stretched or detached. Previously, stretching and detachment had occurred with coatings produced from the same basic formulation, but which had not been subjected to extended sintering times. As previous tests had shown that FEP improved adhesion even when sintered at normal time periods, it would be expected that increased sintering times would have less influence on the adhesion of coatings produced from this bath.

Although the effect of increased adhesion with increased sintering times is again not marked when comparing thin coatings produced from Bath C (i.e. panels #255 and #256), when based on slightly heavier coatings, one can conclude that extended sintering times are beneficial, as previous results with coatings produced from the same bath formulation but not given extended sintering times, resulted in detachment of the coating after only 10 cycles at a much lower load (1,000 grams).

The effect on adhesion of quenching the panels in cold water is not clearly defined. Although results of adhesion tests (employing a knife or thumb nail) had indicated a possible slight loss in adhesion if quenched, NRL tests neither support nor refute this conclusion, as no comparisons can be made at exactly the same film thickness.

As it is unlikely that the film thickness in all areas of a coated part will be in the desired range of approximately 0.0006", it would appear very desirable to employ extended sintering times to insure that heavier coated areas would not become detached. It should also be remembered that even the thin coatings (#255 and #270) that were rated "good" in adhesion within their respective series, were sintered in slight excess of normal.

B. Commercial Possibilities of the Electrodeposition Process

Although most of the work to date has been concerned with electrodeposition of sample panels on a laboratory scale, a number of naval ordnance parts were coated with thin films of TFE by the electrodeposition process. From the results of these experiments it appears that the electrodeposition process possesses a number of advantages over the presently

used spray method. While it is possible to spray TFE by automatic or electrostatic, these methods are little used and the equipment employed is very expensive. Thus any comparisons of the electrodeposition process and spray methods are understood to mean the hand spray method. A general discussion as to the advantages and disadvantages of the electrodeposition process and some of the problems in control follows:

Set-Up Costs - The costs for setting up equipment to coat metal parts with TFE by the electrodeposition process would be nominal. The major expense would be for a DC power source. Due to the short plating cycle and low current densities employed, the power requirements, for coating an equivalent quantity of parts, would be much less than for metal plating. The plating bath, however, is much more expensive and would probably be the limiting factor in scaling up from a pilot plant operation. Although tanks would be required to contain the plating bath, activating tank and rinse tanks, they do not have to be heated, further minimizing set-up costs.

Control of Bath Variables - When both the anode and cathode are immersed in the same plating bath, the pH of the bath will rise during continued use of the bath. Also, the solids content of the bath will decrease. Due to the short plating times and thin coatings applied, the above changes should take place slowly. Thus, these factors can be controlled by the periodic addition of a more concentrated and more acidic bath. The plating bath could also be periodically treated with an ion exchange resin to reduce the pH.

Control of Coating Thickness - The electrodeposition process is capable of producing coatings of extremely accurate average thickness. Better control of thickness than is obtainable by spraying methods should result, as once the conditions are set and provided the bath variables are periodically adjusted, the coating thickness is not dependent on human factors. With respect to the control of thickness on specific areas, more variation is expected as the higher current density areas will receive a heavier coating than the low current density areas. Thickness variations can be minimized by careful cathode design, or by employing current screens, thieving wires, etc., such as are used in chrome plating.

Economics - Economically, electrodeposited TFE coatings should prove advantageous. With this process less material would be used per piece as no material is lost by "overspray". Labor costs should also be less as no time is lost cleaning clogged spray guns, and, most important, many parts can be coated simultaneously.

Quality - The quality of electrodeposited TFE coatings should prove superior to spray coatings with the possible exception of adhesion, where sprayed TFE still has a slight edge. Electrodeposited coatings are smoother and exhibit less surface defects such as blisters, pits, bare spots, coagulated particles, etc.

Shapes - There are certain shapes that are impossible to coat by spray methods that are coatable by electrodeposition methods, such as the inside of small diameter tubing. Although most shapes can be coated by both methods, usually one method possesses a distinct advantage in the coating of a particular shape. The electrodeposition process appears to have the advantage from either a quality or economic standpoint in the coating of the following shapes; illustrations of which are shown on the next page. All items illustrated are coated with TFE and most were coated by the electrodeposition method.

34.

Configuration of Article	Examples
Tubular (coat inside or outside)	#1
Tubular (coat inside and outside simultaneously)	#1, 2
Rod shaped articles with projections or slight recesses	#3, 4, 5, 6
Articles with considerable open areas	#7, 8
Complex four-sided articles	#9, 10
Complex articles with shallow open holes	#11
Thin, narrow articles	#12, 13
Small many faced articles	#14, 15

#1   #2   #3   #4   #5   #6   #7

#8   #9   #10   #11   #12   #13   #14   #15
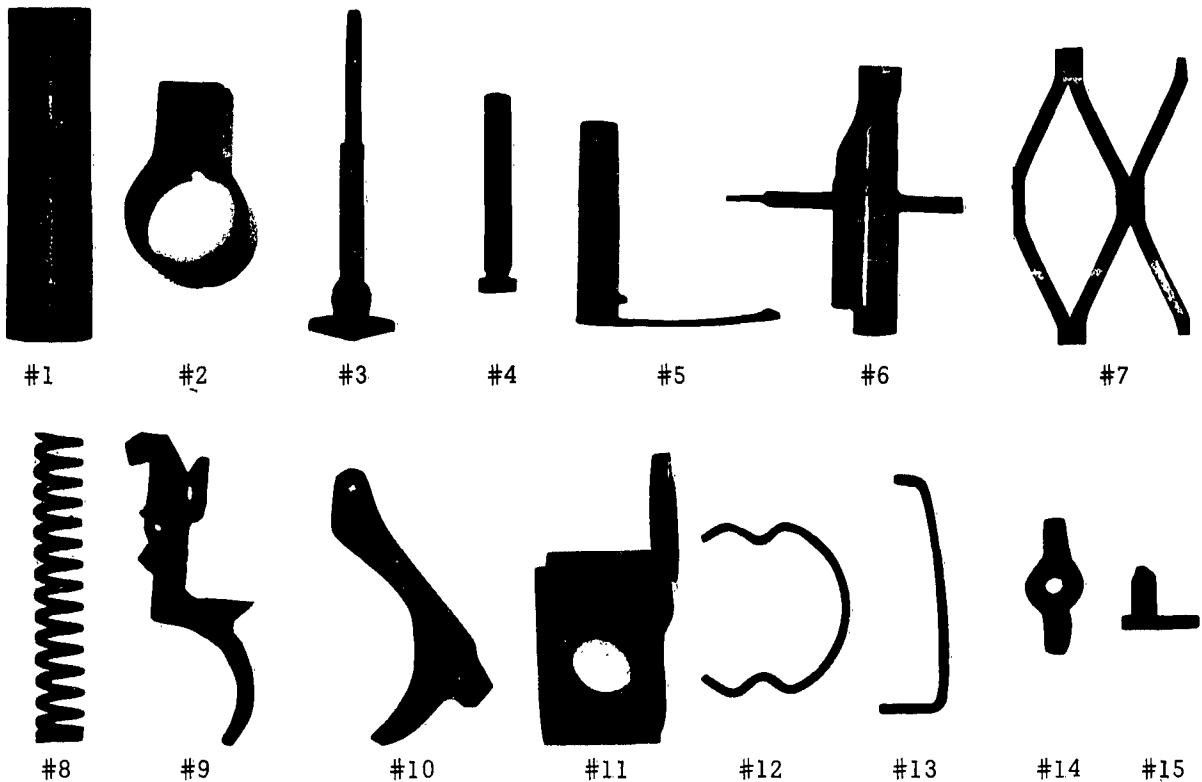
Limitations - The main limitation of the electrodeposition process is that only those metals which are anodically corrodible and can withstand the 700°-750°F. sintering temperature can be coated. This eliminates aluminum, stainless steel and zinc, among others. Due to the considerable trial and error necessary to determine the correct racking set-up and cathode configuration (except for simple shapes), a fair volume of parts of the same shape is required if the use of the process is to be economically justified. Articles that have deep closed recesses or show a large variation in the distance from specific points on the article to the cathode may be difficult to coat.

C. Conclusions.

The electrodeposition method of applying thin coatings of TFE should prove a valuable adjunct to the hand spray method of application. It should find use in dry lubricant applications for the coating of small complex shaped articles when the volume of parts involved is large.

35.

IV. BIBLIOGRAPHY

(1) Thompson, J. B., Turrel, J. C., and Sandt, B. S., "The Coefficient of Friction of Steel on Tetrafluoroethylene Resin", Society of the Plastics Engineers Journal, Vol. 11, No. 4, April, 1955.

(2) Doban, R.C., Sperati, C.A., and Sandt, B.W., "The Physical Properties of 'Teflon' Polytetrafluoroethylene", Society of Plastics Engineers Journal, Vol. 11, No. 9, November, 1955.

(3) Fitzsimmons, V.G., and Zisman, W.A., "Thin Films of Polytetrafluoroethylene Resin (Teflon) as Lubricants and Preservative Coatings for Metals", NRL Report 4753, June 15, 1956.

(4) Davey, W.P., "Process for Making and Applying Japan", U.S. Patent 1,294,627.

(5) Sheppard, S.E., "Electrodeposition of Rubber Coatings", U.S. Patent 1,476,374.

(6) Klein, P., and Szegvari, A., "Method for the Production of Rubber Goods Directly from Latex", U.S. Patent 1,825,736.

(7) Sheppard, S.E., and Eberlin, L.W., "The Electrodeposition of Rubber", Ind. Eng. Chem. 17,711-14 (1925).

(8) Klein, P., "Process and Device for Direct Production of Rubber Sheet and Molded Articles from Latex", U.S. Patent 1,548,689.

(9) Beal, C.L., "Anode Process for Rubber Particles and Coatings", Ind. Eng. Chem., 25,609 (1933).

(10) Marchionna, F., "Latex and Its Industrial Applications", The Rubber Age Publishing Co., New York (1933).

(11) Noble, R.J., "Latex in Industry", The Rubber Age Publishing Co., New York (1953).

(12) Linden, E.G. and Townsend, P., "Encapsulationof Electronic Components and Circuits", Proj. 2005, No5-Materials Engineering Laboratory, Fort Monmouth, N.J.

(13) Feinleib, M., Trans. Electrochemical Soc., Vol. 88, 11-23 (1945).

(14) Logan, R.W., and Nelson, M.C., General Plastics Corporation, unpublished data.

(15) Gray, A.G., "Electrophoretic Deposition of Ethylene Polymers", U.S.Patent 2,530,366.

(16) Eberlin, L.W., and Beal, C.L., "Electrodeposition of Coatings of Cellulosic Compounds", U.S. Patent 1,589,327.

(17) Hochberg, S., "Polytetrafluoroethylene Coating Compositions", U.S.Patent,2,681,324.

(18) Heller, G.W., "Electrodeposition of Tetrafluoroethylene Polymers", U.S. Patent 2,562,117.

(19) Du Pont Information Bulletin "Teflon 41BX-Aqueous Dispersion Properties and Processing Techniques", Polychemicals Department, E. I. Du Pont.

(20) "An Electrodeposition Process for Applying Coatings of 'Teflon' Polytetrafluoroethylene Resins to Electroconductive Surfaces", Marketing Section, Polychemicals Department, E. I. Du Pont.

(21) "Electrodeposition of Teflon on Complex Shapes", E.I. Du Pont Sales Service Laboratory, Polychemicals Department (SSL #225T)

(22) Logan, R.W., "Research and Development of Thin Polytetrafluoroethylene (Teflon) Coatings by Electrodeposition", Contract No. Nord-18845, Dept. of the Navy, Bureau of Ordnance, Final Report, 1 June, 1959 - 1, June, 1960.

(23) Logan, R.W., "Research & Development of Thin Polytetrafluoroethylene (Teflon) Coatings by Electrodeposition", Contract No. Now-60-0755C, Dept. of the Navy, Bureau of Naval Weapons, Final Report, 1 July, 1960 - 1 July, 1961.

(24) Bernett, M.K. and Zisman, W. A., "Synergistic Surface Tension Effects from Mixtures of Fluorinated Alcohols with Conventional Wetting Agents", NRL Report 5556, October 20, 1960.

(25) Fisher, E.K., and Gans, D.M., Ann. N.Y. Acad. Sci. 46:371 (1946)

(26) Bernett, M.K., and Zisman, W.A., "Wetting of Low-Energy Solids by Aqueous Solutions of Highly Fluorinated Acids and Salts", J. Phys. Chem. 63:1241 (1959).

(27) Bernett, M.K. and Zisman, W.A. "Synergistic Surface Tension Effects from Mixtures of Fluorinated Alcohols with Conventional Wetting Agents", NRL Report 5556, October 20, 1960.

(28) " 'Cassel' Surface Tensiometer", Product Literature, National Instrument Laboratories Inc., Washington, D.C.

(29) Cassel, H.M., U.S. Patent 2,448,768, Sept. 7, 1948.

(30) Osdal, L.K., "Polytetrafluoroethylene Coating Compositions", U.S. Patent, 2,562,118.

(31) Osdal, L.K., "Polytetrafluoroethylene Coating Compositions", U. S. Patent, 2,562,117.

(32) Personal communication from V. G. Fitzsimmons, NRL, Washington, D. C., 12 June, 1961. (Examination of Electrodeposited TFE Coatings.)

(33) Cahne, A., "Method of Coating a Metal Base with Polytetrafluoroethylene, U. S. Patent 2,944,917.

TABLE I

Bath Formula - 30.0% solids TEFLON 41BX
                 3.0% sodium diamyl sulfosuccinate (based on the TFE weight)
Conductivity = 1,750/70°F. micromhos   pH = 6.2

Panel	Etch	V	A	CD	T	Coating Thickness Mil-calc.	Cracks(24X)	Appearance of deposit(24X)	Adhesion lbs/in.	Final Thick (mil)
1	Yes	16	.2	07.7	3.0	0.39	Yes (10%)	Good	1.2-1.2	2.6
2	Yes	28	.4	15.4	3.0	0.92	Yes (40%)	Good	1.2-1.2	2.6
3	Yes	40	.6	23.0	2.0	1.27	Yes (100%)	Good	1.5-1.5	2.9
4	Yes	36	.5	19.2	1.5	0.55	Yes (10%)	Good	1.8-1.6	2.5

Bath Formula - 30.0% solids TEFLON 41BX
                 3.0% sodium diamyl sulfosuccinate (based on the TFE weight)
                10.0% chrome oxide green solids (based on the TFE weight)
Conductivity = 2,375/70°F. micromhos

Panel	Etch	V	A	CD	T	Coating Thickness Mil-calc.	Cracks(24X)	Appearance of deposit(24X)	Adhesion lbs/in.	Final Thick (mil)
1	Yes	32	.5	19.2	2.0	0.43	Yes (10%)	Poor-thin areas	1.3-1.2	2.0
2	Yes	32	.5	19.2	2.0	0.38	Yes (10%)	Poor-thin areas	1.8-1.4	2.3
3	Yes	32	.5	19.2	2.0	0.47	Yes (10%)	Poor-thin areas	----	---
4	Yes	32	.5	19.2	3.0	0.56	Yes (30%)	Poor-thin areas	----	---
5	Yes	48	.7	26.9	2.0	0.51	Yes (30%)	Poor-thin areas	1.2-1.2	2.0
6	Yes	14	.2	7.7	6.0	0.41	Yes (10%)	Poor-thin areas	1.4-1.0	2.2

Bath Formula - 30.0% solids TEFLON 41BX
                 3.0% sodium diamyl sulfosuccinate (based on the TFE weight)
                20.0% chrome oxide green solids (based on the TFE weight)
Conductivity = 2,600/70°F. micromhos

Panel	Etch	V	A	CD	T	Coating Thickness Mil-calc.	Cracks(24X)	Appearance of deposit(24X)	Adhesion lbs/in.	Final Thick (mil)
1	Yes	30	.5	19.2	3.0	0.36	No	Poor-thin areas*	----	---
2	Yes	30	.5	19.2	6.0	0.51	Yes (10%)	Poor-thin areas*	----	---

TABLE II

Bath Formula - 30.0% solids TEFLON 41BX
                 3.0% sodium dioctyl sulfosuccinate (based on the TFE weight)
Conductivity = 1,440/70°F. micromhos   pH = 6.4

Panel	Etch	V	A	CD	T	Coating Thickness Mil-calc.	Cracks(24X)	Appearance of deposits(24X)	Adhesion lbs./in.	Final Thick (mil)
1	Yes	50	.5	19.2	3.0	1.60	Yes (100%)	Excellent	1.2-1.2	3.0
2	Yes	50	.5	19.2	2.0	1.12	Yes (100%)	Excellent	2.2-1.6	2.3
3	Yes	50	.5	19.2	1.0	0.70	Yes (20%)	Excellent	2.2-1.4	2.8
4	Yes	50	.5	19.2	1.0	0.70	Yes (10%)	Excellent	1.6-1.0	2.0

Bath Formula - 30.0% solids TEFLON 41BX
                 3.0% sodium dioctyl sulfosuccinate (based on the TFE weight)
                20.0% chrome oxide green solids (based on the TFE weight)
Conductivity = 1,900/70°F. micromhos

Panel	Etch	V	A	CD	T	Coating Thickness Mil-calc.	Cracks(24X)	Appearance of deposits(24X)	Adhesion lbs./in.	Final Thick (mil)
1	Yes	40	.5	19.2	1.0	0.46	Yes (10%)	Fair-thin areas	1.8-1.4	2.8
2	Yes	40	.5	19.2	1.0	0.42	Yes (10%)	Fair-thin areas	1.8-1.2	2.5
3	Yes	40	.5	19.2	1.5	0.63	Yes (10%)	Fair-thin areas	1.6-1.2	2.4
4	Yes	40	.5	19.2	2.0	0.84	Yes (30%)	Good	1.6-1.0	2.6
5	Yes	40	.5	19.2	2.0	0.82	Yes (30%)	Good	1.6-1.0	2.5
6	Yes	34	.5	19.2	2.0	0.89	Yes (50%)	Excellent	1.8-1.4	2.8

*Pigment Flocculated

38.

TABLE III

Adhesion of Electrodeposited TFE Coatings

Bath Formula - 30.0% solids TEFLON 41BX
　　　　　　　　3.0% sodium dihexyl sulfosuccinate (based on the TFE weight)
Conductivity ≈ 1,740/70°F. micromhos　　　pH = 6.0

Panel	Etch	V	A	CD	T	Coating Thickness Mil-calc.	Cracks (24X)	Appearance of deposit (24X)	Adhesion lbs/in.	Final Thick (mil)
1	Yes	40	.5	19.2	3.0	1.56	Yes (50%)	Good	---	---
2	Yes	40	.5	19.2	3.0	1.80	Yes (70%)	Good	1.0-1.0	3.0
3	Yes	40	.5	19.2	2.0	0.90	Yes (30%)	Good	0.8-0.8	2.2

Bath Formula - 30.0% solids TEFLON 41BX
　　　　　　　　3.0% sodium dihexyl sulfosuccinate (based on the TFE weight)
　　　　　　　　10.0% chrome oxide green solids (based on the TFE weight)
Conductivity = 1,900/70°F. micromhos

Panel	Etch	V	A	CD	T	Coating Thickness Mil-calc.	Cracks (24X)	Appearance of deposit (24X)	Adhesion lbs/in.	Final Thick (mil)
1	Yes	34	.5	19.2	3.0	0.90	Yes (50%)	Fair	1.0-1.0	2.0
2	Yes	34	.5	19.2	3.0	0.88	Yes (50%)	Good	1.0-1.0	2.1
3	Yes	34	.5	19.2	6.0	1.49	Yes (90%)	Good	---	---

Bath Formula - 30.0% solids TEFLON 41BX
　　　　　　　　3.0% sodium dihexyl sulfosuccinate (based on the TFE weight)
　　　　　　　　20.0% chrome oxide green solids (based on the TFE weight)
Conductivity = 2,350/70°F. micromhos

Panel	Etch	V	A	CD	T	Coating Thickness Mil-calc.	Cracks (24X)	Appearance of deposit (24X)	Adhesion lbs/in.	Final Thick (mil)
1	Yes	32	.5	19.2	3.0	0.53	Yes (10%)	Fair-thin areas	---	---
2	Yes	32	.5	19.2	2.5	0.38	Yes (10%)	Fair-thin areas	---	---
3	Yes	32	.5	19.2	4.0	0.56	Yes (10%)	Fair-thin areas	1.4-1.2	2.5

Bath Formula - 30.0% solids TEFLON 41BX
　　　　　　　　3.0% sodium dihexyl sulfosuccinate (based on the TFE weight)
　　　　　　　　40.0% chrome oxide green solids (based on the TFE weight)
Conductivity = 2,700/70°F. micromhos

Panel	Etch	V	A	CD	T	Coating Thickness Mil-calc.	Cracks (24X)	Appearance of deposit (24X)	Adhesion lbs/in.	Final Thick (mil)
1	Yes	30	.5	19.2	4.0	0.83	Yes (60%)	Poor-thin areas*	1.0-0.8	2.5
2	Yes	28	.5	19.2	6.0	1.19	Yes (40%)	Fair-thin areas*	1.0-0.6	2.8

Bath Formula - 30.0% solids TEFLON 41 BX
　　　　　　　　3.0% sodium dihexyl sulfosuccinate (based on the TFE weight)
　　　　　　　　60.0% chrome oxide green solids (based on the TFE weight)
Conductivity = 3,150/70°F. micromhos

Panel	Etch	V	A	CD	T	Coating Thickness Mil-calc.	Cracks (24X)	Appearance of deposit (24X)	Adhesion lbs/in.	Final Thick (mil)
1	Yes	24	.5	19.2	6.0	1.16	Yes (30%)	Poor-thin areas*	1.2-1.0	2.4
2	Yes	24	.5	19.2	4.0	0.73	Yes (10%)	Fair-thin areas*	1.0-1.0	2.5

*Pigment flocculates on drying.

TABLE IV
Adhesion of Electrodeposited TFE Coatings

Bath Formula - 30.0% solids TEFLON 41BX
      6.0% sodium dihexyl sulfosuccinate(based on the TFE weight)
      30.0% ∅ C_6 alcohol (based on the weight of the sodium dihexyl
            sulfosuccinate)
Conductivity = 2,800/70°F. micromhos   pH = 5.7

Panel	Etch	V	A	CD	T	mil-calc.	Cracks(24X)	Appearance of deposit(24X)	Adhesion lbs./in.	Final Thick (mil)
1	Yes	28	.5	19.2	1.0	0.49	Yes (30%)	Fair-streaked	1.4-1.0	2.4
2	Yes	28	.5	19.2	2.0	0.60	Yes (70%)	Fair-streaked	1.4-1.2	2.5
3	Yes	24	.5	19.2	3.0	1.00	Yes (10%)	Fair-streaked	1.0-0.9	2.4
4	Yes	24	.5	19.2	4.0	1.32	Yes (30%)	Fair-streaked	1.0-0.8	3.8
5	Yes	24	.5	19.2	3.0	0.85	Yes (10%)	Fair-streaked	---	---

Bath Formula - 30.0% solids TEFLON 41BX
      6.0% sodium dihexyl sulfosuccinate (based on the TFE weight)
      30.0% ∅ C_6 alcohol (based on the weight of the sodium dihexyl
            sulfosuccinate)
      10.0% chrome oxide green solids (based on the TFE weight)
Conductivity = 3,000/70°F. micromhos

Panel	Etch	V	A	CD	T	mil-calc.	Cracks(24X)	Appearance of deposit(24X)	Adhesion lbs./in.	Final Thick (mil)
1	Yes	24	.5	19.2	3.0	0.85	Yes (60%)	Poor*	1.2-1.2	2.1

*Pigment flocculated.

TABLE V

Adhesion of Electrodeposited TFE Coatings

Bath Formula - 30.0% solids TEFLON 41BX
      3.0% "Zonyl" S-13 (based on the TFE weight)
Conductivity = 1,350/70°F. micromhos   pH = 4.0

Panel	Etch	V	A	CD	T	Coating Thickness mil-calc.	Cracks(24X)	Appearance of deposit(24X)	Adhesion lbs./in.	Final Thick (mil)
1	Yes	42	.5	19.2	4.0	2.60	Yes (100%)	Poor-V.rough	---	---
2	Yes	42	.5	19.2	2.0	1.50	Yes (100%)	Poor-V.rough	2.2-1.8	3.5
3	Yes	42	.5	19.2	1.0	0.84	Yes (100%)	Poor-V.rough	2.4-2.0	3.2
4	Yes	42	.5	19.2	0.8	0.72	Yes (100%)	Poor-V.rough	2.2-1.6	2.9

Bath Formula - 30.0% solids TEFLON 41BX
      3.0% "Zonyl" S-13 (based on the TFE weight)
      20.0% chrome oxide green solids (based on the TFE weight)
Conductivity = 1.980/70°F. micromhos

Panel	Etch	V	A	CD	T	mil-calc.	Cracks(24X)	Appearance of deposit	Adhesion lbs./in.	Final Thick (mil)
1	Yes	32	.5	19.2	1.0	0.66	Yes (10%)	Fair-rough	2.4-1.8	2.5
2	Yes	32	.5	19.2	2.0	0.98	Yes (20%)	Fair-rough	2.8-2.4	3.9
3	Yes	32	.5	19.2	2.0	1.02	Yes (40%)	Fair-rough	2.6-2.2	3.4
4	Yes	32	.5	19.2	1.5	0.85	Yes (20%)	Fair-rough	2.4-2.0	3.0

TABLE VI

Adhesion of Electrodeposited TFE Coatings

Bath Formula - 30.0% solids TEFLON 41BX
        3.0% ø octanoic acid (based on the TFE weight)
        20.0% chrome oxide green solids (based on the TFE weight)
Conductivity = 1,950/70°F. micromhos   pH = 4.5

Panel	Etch	V	A	CD	T	Coating Thickness Mil-calc.	Cracks(24X)	Appearance of deposit(24X)	Adhesion lbs./in.	Final Thick (mil)
1	Yes	32	.5	19.2	3.0	1.23	No	Excellent	----	---
2	Yes	36	.5	19.2	2.0	0.91	No	Excellent	3.0-3.0*	2.2
3	Yes	36	.5	19.2	2.0	0.80	No	Excellent	1.4-1.2	2.3
4	Yes	36	.5	19.2	2.0	0.80	No	Good	2.4-2.2	2.3
5	Yes	36	.5	19.2	2.0	0.79	No	Good	3.4-3.4**	2.1
6	Yes	36	.5	19.2	1.8	0.73	No	Good	---	---
7	Yes	36	.5	19.2	1.8	0.71	No	Good	---	---

*Film broke - Built up to 3.7 mil - Adhesion = 2.4-2.0
**Film broke - Built up to 4.0 mil - Adhesion = 2.6-2.5

Bath Formula - 30.0% solids TEFLON 41BX
        3.0% ø octanoic acid (based on the TFE weight)
        40.0% chrome oxide green solids (based on the TFE weight)
Conductivity = 2,300/70°F. micromhos

Panel	Etch	V	A	CD	T	Coating Thickness Mil-calc.	Cracks(24X)	Appearance of deposit(24X)	Adhesion lbs./in.	Final Thick (mil)
1	Yes	36	.5	19.2	2.0	0.80	No	Excellent	1.6-1.4	2.5
2	Yes	36	.5	19.2	2.0	0.82	No	Excellent	2.6-2.2	2.5
3	Yes	36	.5	19.2	2.0	0.84	No	Excellent	1.2-1.0	2.5
4	Yes	36	.5	19.2	2.0	0.80	No	Excellent	1.2-1.0	2.5
5	Yes	32	.5	19.2	2.0	0.83	No	Excellent	1.5-1.4	3.0
6	Yes	32	.5	19.2	2.0	0.78	No	Excellent	2.3-2.0	2.5
7	Yes	38	.5	19.2	3.0	1.12	No	Excellent	1.5-1.3	3.2
8	Yes	38	.5	19.2	3.0	1.18	No	Excellent	1.3-1.0	3.0

Bath Formula - 30.0% solids TEFLON 41BX
        3.0% ø octanoic acid (based on the TFE weight)
        60.0% chrome oxide green solids (based on the TFE weight)
Conductivity = 2,580/70°F. micromhos

Panel	Etch	V	A	CD	T	Coating Thickness Mil-calc.	Cracks(24X)	Appearance of deposit(24X)	Adhesion lbs./in.	Final Thick (mil)
1	Yes	36	.5	19.2	2.0	0.63	No	Good-sl.pig.floc.	2.6-2.0	2.2
2	Yes	24	.5	19.2	3.0	1.32	Yes (20%)	Good-sl.pig.floc.	3.0-3.0***	2.1
3	Yes	24	.5	19.2	2.0	0.64	No	Good-sl.pig.floc.	1.3-1.2	1.6
4	Yes	30	.5	19.2	2.0	0.67	No	Good-sl.pig.floc.	1.7-1.5	1.8
5	Yes	30	.5	19.2	2.0	0.61	No	Good-sl.pig.floc.	2.1-1.9	1.8
6	Yes	30	.5	19.2	2.0	0.66	No	Good-sl.pig.floc.	1.7-1.5	1.7

***Film broke after partially stripped.

TABLE VII

Bath Formula - 30.0% solids TEFLON 41BX
   3.0% ∅ octanoic acid (based on the TFE weight)
   20.0% chrome oxide green solids (based on the TFE weight)
   20.0% FEP (based on the TFE weight) - #856-200 Finishes Div.
Conductivity = 1,900/70°F. micromhos

Panel	Etch	V	A	CD	T	Coating Thickness mil-calc.	Cracks(24X)	Appearance of deposit(24X)	Adhesion lbs./in.	Final Thick (mil)
1	Yes	40	.5	19.2	2.0	0.68	Yes (10%)	Fair-pig.floc.	1.4-1.0	2.3
2	Yes	40	.5	19.2	4.0	1.06	Yes (100%)	Fair-pig.floc.	1.4-1.3	2.8
3	Yes	40	.5	19.2	3.0	0.80	Yes (40%)	Fair-pig.floc.	1.2-1.0	3.7
4	Yes	40	.5	19.2	2.0	0.72	Yes (20%)	Fair-pig.floc.	1.2-1.0	2.9

Bath Formula - 30.0% solids TEFLON 41BX
   4.0% ∅ octanoic acid (based on the TFE weight)
   20.0% chrome oxide green solids (based on the TFE weight)
   20.0% FEP (based on the TFE weight) - #856-200 Finishes Div.
Conductivity = 1,950/70°F.

1	Yes	34	.5	19.2	2.0	1.01	Yes (20%)	Good-sl.pig.floc.	1.0-0.9	3.2
2	Yes	34	.5	19.2	1.2	0.75	Yes (20%)	Good-sl.pig.floc.	2.6-2.2	2.9
3	Yes	40	.5	19.2	2.0	0.98	Yes (30%)	Good-sl.pig.floc.	2.5-2.3	2.3
4	Yes	40	.5	19.2	1.5	0.73	Yes (10%)	Good-sl.pig.floc.	2.0-1.7	2.1
5	Yes	40	.5	19.2	1.5	0.75	Yes (20%)	Good-sl.pig.floc.	2.4-2.0	2.2
6	Yes	40	.5	19.2	1.5	0.77	Yes (10%)	Good-sl.pig.floc.	2.3-2.0	2.2
7	Yes	40	.5	19.2	1.5	0.77	Yes (10%)	Good-sl.pig.floc.	2.3-2.1	1.8

TABLE VIII

30.0% TEFLON 41 BX
   3.0% ∅ octanoic acid (based on the TFE weight)
   10.0% alumina modified colloidal silica (based on the TFE weight)
   pH = 4.0   Conductivity = 1,720/70°F. micromhos

No.	Etch	V	A	CD	T	Thickness mil. -calc.	Cracks(24X)	Appearance (24X)	Adhesion	Thick (mil)
49	Yes	32	.5	19.2	2.0	0.88	Yes (60%)	Good	4.6-4.4	2.5
50	Yes	32	.5	19.2	1.0	0.47	Yes (40%)	Good	4.6-4.4	2.5
8	Yes	32	.5	19.2	1.5	0.68	Yes (30%)	Good	3.2-2.8	2.7
53	Yes	32	.5	19.2	1.5	0.68	Yes (20%)	Good	3.5-3.0	2.8
46	Yes	32	.5	19.2	1.0	0.61	Yes (10%)	Good	2.5-2.0	2.5
44	Yes	32	.5	19.2	0.5	0.31	No	Poor	--	--
51	Yes	34	.5	19.2	1.0	0.60	Yes (10%)	Good	3.1-3.8	2.5
4	Yes	34	.5	19.2	1.0	0.63	Yes (10%)	Good	2.5-2.0	3.1
1	Yes	34	.5	19.2	0.5	0.35	No	Fair	2.6-2.4	2.4
21	Yes	34	.5	19.2	0.5	0.35	No	Fair	4.0+	3.7
12	Yes	34	.5	19.2	1.0	0.74	Yes (30%)	Good	2.0-1.5	2.8
11	Yes	38	.5	19.2	1.0	0.72	No	Good	2.2-1.8	2.8
13	Yes	38	.5	19.2	1.0	0.72	Yes(5%)	Good	2.8-2.4	3.0
14	No	38	.5	19.2	1.0	0.42	No	Poor	2.4-1.8	2.7
15	No	38	.5	19.2	1.0	0.36	No	Poor		
16	No	38	.5	19.2	2.0	0.61	Yes(5%)	Poor	3.2-2.2	2.5

TABLE IX

Adhesion Of Miscellaneous TFE Coatings to Steel Substrates Pretreated With HINAC I-X*

Formula – HINAC A-2 = 24.0 cc
HINAC R-3 = 7.7 cc
Made up to a total volume of 500 cc's with tap water.

Procedure – SAE 1010 steel panels were sandblasted with #80 silica sand and dipped briefly (10 seconds) in the above HINAC I-X solution, allowed to drain, and baked for ten minutes at 400°F. The HINAC solution was maintained at room temperature except for panel #10, which was dipped in the HINAC solution at 140°F. The TFE coatings were then applied by the method shown, preceded by a 10% HCL etch (or activation step) where noted. The TFE panels were built up further by topcoating (by spraying) with TFE and the adhesion checked.

Panel	No. of dips in HINAC I-X	Prime Coat Application	Baking Schedule	Appearance of TFE Coating	Top Coat(s)	Adhesion lbs./in.	Final Thick (mil)
1	2	Sprayed #852-201 (1 ct.)	10'/750°F.	Good-sl. rust spots	#851-224	1.0-0.6	1.3
2	1	Sprayed #852-201 (1 ct.)	10'/750°F.	Fair-medium rust spots	#851-224	1.0-0.6	1.7
3	1	Sprayed #851-224 (1 ct.)	10'/750°F.	Poor-heavy rust spots	None	0.5-0.4	1.3
4	1	Sprayed Formula A (1 ct.)	10'/750°F.	Fair-cracked-streaked black	#851-224	3.6-3.2	2.8
5	1	Sprayed Formula A(2 cts)	10'/750°F.	Good-cracked	#851-224	1.6-1.4	1.8
6	1	Sprayed 63%FEP** (1 ct.)	10'/750°F.	Good-discontinuous film	#851-224	2.0-1.8	2.0
7	1	Sprayed Formula B (1 ct.)	10'/750°F.	Fair-streaked black	#851-224	1.7-1.5	1.7
8	1	Plated Formula C (1 ct.)	10'/750°F.	Poor-blisters, bare spots	#851-224	0.8-0.6	2.1
9	1	Plated Formula C***(1ct.)	10'/750°F.	Good-slight blisters	#851-224	1.8-1.4	1.3
10	1	Sprayed #852-201 (1 ct)	10'/750°F.	Good-slight rust spots	#851-224	1.0-0.6	1.9

*Registered Trademark, Pennsalt Chemicals

**Manufacturer – Du Pont Plastics Division – #TE-9500

***Acid etched with 10% HCL before plating at room temperature for 5 seconds.

Formula A
30.0% solids TEFLON 41BX
3.4% ∅ octanoic acid (based on the TFE wt)
150.0% FEP solids (based on the TFE weight)
(added at 63% solids)

Formula B
35.0% solids FEP (DuPont-Plastics Div. #TE-9500)
3.0% ∅ octanoic acid(based on FEP wt.)
2.0% carbon black solids (based on FEP wt)

Formula C
30.0% solids TEFLON 41BX
3.0% ∅ octanoic acid (based on the TFE weight)
20.0% chrome oxide green solids (based on the TFE weight)

43.

TABLE IX

TABLE X

Adhesion of TFE Coatings on Chemically Etched Steel

Panels - SAE 1010 Steel

Series A

Procedure - Panels were sandblasted(#80 silica sand) and etched in 10% nitric acid solution, then coated with TFE

Panel	HNO₃ Etching time(min)	Etching Temp.°F.	Prime Coat	Baking Schedule	Appearance(24X)	Top Coat(s)	Adhesion lbs./in.	Final Thick. (mil)
1	10'	88-100	Sprayed#852-201 (1 ct.)	10'/750°F.	Good-sl.rust spots	#851-224	1.8-1.2	4.2
2	10'	88-100	Dipped Teflon30(>42.6)(lct.)	10'/750°F.	Fair-streaked	#851-224	1.8-1.0	3.8
3	10'	88-100	Sprayed #851-224 (1 ct.)	10'/750°F.	Poor-heavy rust spots	#851-224	1.6-1.2	4.2
4	10'	88-100	Sprayed Formula A (1 ct.)	10'/750°F.	Fair-streaked black	#851-224	2.8-2.4	3.6
5	10'	88-100	Sprayed FEP 63%(TE-9500)(ct)	10'/750°F.	Good-discontinuous	#851-224	2.9-2.5	5.3
6	10'	90-102	Sprayed Formula B(1 ct.)	10'/750°F.	Fair-streaked black	#851-224	4.4-4.2	4.7
7	10'	90-102	Sprayed FEP #856-200(1 ct.)	10'/750°F.	Good-sl. cracked	#851-224	4.0-3.2	5.0
8	10'	90-102	Plated Formula C*(1 ct.)	10'/750°F.	Poor-blisters & pits	#851-224	1.6-0.6	5.2
9	10'	90-102	Dipped#3170(>30%)(1 ct.)	10'/750°F.	Good-sl. streaks	#851-224	0.7-0.5	(2.2)
10	10'	90-102	Dipped#3170 (60%) (1 ct.)	10'/750°F.	Fair-streaked	#851-224	0.3-0.4	(3.0)

Series B

Procedure - Panels were sandblasted and then etched in 10% nitric acid solution containing 0.1% of a fluorochemical wetting agent (3M Co. - FC-95)

Panel	HNO₃ Etching time(min)	Etching Temp.°F.	Prime Coat	Baking Schedule	Appearance(24X)	Top Coat(s)	Adhesion lbs./in.	Final Thick. (mil)
1	10'	92-98	Sprayed #852-201 (1 ct.)	10'/750°F.	Good-sl.rust spots	#851-224	2.6-1.8	4.0
2	10'	92-98	Sprayed TFE 30 (>42.6)(lct.)	10'/750°F.	Fair-streaked	#851-224	2.4-1.6	4.2
3	10'	92-98	Sprayed #851-224 (1 ct.)	10'/750°F.	Poor-heavy rust spots	#851-224	2.6-1.8	5.0
4	10'	92-98	Sprayed Formula A (1 ct.)	10'/750°F.	Fair-streaked black	#851-224	2.2-2.0	4.2
5	10'	92-98	Sprayed 63%FEP(TE-9500)(1ct)	10'/750°F.	Poor-heavy cracks	#851-224	3.6-3.4	3.6
6	10'	92-93	Sprayed Formula-B - (1 ct.)	10'/750°F.	Fair-streaked black	#851-224	5.2-4.8	3.5
7	10'	92-93	Sprayed FEP #856-200(1 ct.)	10'/750°F.	Good-sl. cracked	#851-224	3.2-2.8	4.2
8	10'	92-93	Plated Formula C**(1 ct.)	10'/750°F.	Fair-blisters & Pits	#851-224	1.4-1.0	5.0
9	10'	92-93	Dipped#3170(>30%) (1 ct.)	10'/750°F.	Good-sl. streaked	#851-224	1.0-0.6	(2.3)
10	10'	92-93	Dipped#3170(60%) (1 ct.)	10'/750°F.	Fair-streaks	#851-224	0.7-0.6	(2.9)

*Acid etched with 10% HCL (6 seconds at room temperature) before plating.

**Acid etched with 10% HCL (30 seconds at 150°F.) before plating.

Formula A
30.0% solids TEFLON 41BX
3.4% ø octanoic acid(based on the TFEwt.)
150.0% FEP solids (based on the TFE wt.)
(added as 63% solids (#TE-9500))

Formula B
35.0% solids FEP (#TE-9500)
3.0% ø octanoic acid(based on TFE wt.)
2.0% carbon black solids (based on the FEP weight)

Formula C
30.0% solids TEFLON 41BX
3.0% ø octanoic acid(based on TFE wt)
20.0% chrome oxide green solids (based on the TFE weight)

44.

TABLE XI

Adhesion of Electrodeposited TFE Coatings to Chemically Etched Steel

Bath Formula - 30.0% solids TEFLON 41BX
            3.0% ∅ octanoic acid (based on the TFE weight)
            20.0% chrome oxide green solids (based on the TFE weight)
Conductivity = 1,800/70°F. micromhos

Etchant - 10% HNO_3 - No wetting agent added
Time   - 12 minutes at 80°F.
Metal removed during etch = 0,120 grams/sq. in. (approx.)

Panel	Etch 10%HCL	V	A	CD	T	Coating Thickness mil-calc.	Cracks(24X)	Appearance of deposit(24X)	Adhesion lbs./in.	Final Thick (mil)
1	Yes(a)	36	.5	19.2	2.0	0.76	Yes(20%)	Poor-bare areas	0.4-0.3	2.8
2	Yes(b)	36	.5	19.2	2.0	0.66	Yes(20%)	Poor-bare areas	0.5-0.3	3.0
3	Yes	36	.5	19.2	2.0	0.73	Yes(10%)	Poor-bare areas	0.4-0.4	2.9
4	Yes	36	.5	19.2	2.0	0.64	Yes(20%)	Poor-bare areas	0.4-0.3	2.8
5	Yes(c)	36	.5	19.2	2.0	0.79	Yes(10%)	Good	1.8-1.6	3.0
6	Yes(c)	36	.5	19.2	1.5	0.61	No	Good	1.6-1.4	3.2

Bath Formula - 30.0% solids TEFLON 41BX
            4.0% ∅ octanoic acid (based on the TFE weight)
            20.0% chrome oxide green solids (based on the TFE weight)
            20.0% FEP(based on the TFE weight) -(#856-200 DuPont Finishes
Conductivity = 1,950/70°F. micromhos   Div.)

Etchant - 10% HNO_3 - No wetting agent added
Time   - 12 minutes at 80°F.- 80°F.
Metal removed during etch = 0.120 grams/sq. in. (approx.)

1	Yes	40	.5	19.2	1.5	0.59	Yes(10%)	Poor-blisters-bare areas	2.4-1.8	(2.1)
2	Yes	40	.5	19.2	1.5	0.62	Yes(20%)	Poor-blisters-bare areas	2.2-1.8	(2.2)
3	Yes	40	.5	19.2	1.5	0.59	Yes(20%)	Poor-blisters-bare areas	2.2-1.6	(2.3)
4	Yes(c)	40	.5	19.2	1.5	0.65	Yes(10%)	Fair-thin areas-pig.floc.	2.2-1.6	(2.2)
5	Yes(c)	50	.5	19.2	1.5	0.66	Yes(10%)	Fair-thin areas-pig.floc.	2.2-1.8	(2.0)
6	Yes(c)	40	.5	19.2	1.5	0.63	Yes(10%)	Fair-thin areas-pig.floc.	2.0-1.8	(2.2)

Notes:
(a) Etched in 10% HCL at room temperature for 30 seconds
(b) Etched in 10% HCL at 200°F. for 30 seconds
(c) Sandblasted with #80 silica sand following the nitric acid etch and then activated with 10% HCL at the normal time and temperature of 5 seconds at 75°F.

TABLE XI

Adhesion of Electrodeposited TFE Coatings to Chemically Etched Steel

Bath Formula - 30.0% solids TEFLON 41BX
       3.0% ∅ octanoic acid (based on the TFE weight)
      20.0% chrome oxide green solids (based on the TFE weight)
Conductivity = 1,800/70°F. micromhos

Etchant - 10% HNO_3 - No wetting agent added
Time   - 12 minutes at 80°F.
Metal removed during etch = 0.120 grams/sq. in. (approx.)

Panel	Etch 10%HCL	V	A	CD	T	Coating Thickness mil-calc.	Cracks(24X)	Appearance of deposit(24X)	Adhesion lbs./in.	Final Thick (mil)
1	Yes(a)	36	.5	19.2	2.0	0.76	Yes(20%)	Poor-bare areas	0.4-0.3	2.8
2	Yes(b)	36	.5	19.2	2.0	0.66	Yes(20%)	Poor-bare areas	0.5-0.3	3.0
3	Yes	36	.5	19.2	2.0	0.73	Yes(10%)	Poor-bare areas	0.4-0.4	2.9
4	Yes	36	.5	19.2	2.0	0.64	Yes(20%)	Poor-bare areas	0.4-0.3	2.8
5	Yes(c)	36	.5	19.2	2.0	0.79	Yes(10%)	Good	1.8-1.6	3.0
6	Yes(c)	36	.5	19.2	1.5	0.61	No	Good	1.6-1.4	3.2

Bath Formula - 30.0% solids TEFLON 41BX
       4.0% ∅ octanoic acid (based on the TFE weight)
      20.0% chrome oxide green solids (based on the TFE weight)
      20.0% FEP(based on the TFE weight) -(#856-200 DuPont Finishes
Conductivity = 1,950/70°F. micromhos   Div.)

Etchant - 10% HNO_3 - No wetting agent added
Time   - 12 minutes at 80°F.- 80°F.
Metal removed during etch = 0.120 grams/sq. in. (approx.)

1	Yes	40	.5	19.2	1.5	0.59	Yes(10%)	Poor-blisters-bare areas	2.4-1.8	(2.1)
2	Yes	40	.5	19.2	1.5	0.62	Yes(20%)	Poor-blisters-bare areas	2.2-1.8	(2.2)
3	Yes	40	.5	19.2	1.5	0.59	Yes(20%)	Poor-blisters-bare areas	2.2-1.6	(2.3)
4	Yes(c)	40	.5	19.2	1.5	0.65	Yes(10%)	Fair-thin areas-pig.floc.	2.2-1.6	(2.2)
5	Yes(c)	50	.5	19.2	1.5	0.66	Yes(10%)	Fair-thin areas-pig.floc.	2.2-1.8	(2.0)
6	Yes(c)	40	.5	19.2	1.5	0.63	Yes(10%)	Fair-thin areas-pig.floc.	2.0-1.8	(2.2)

Notes:
(a) Etched in 10% HCL at room temperature for 30 seconds
(b) Etched in 10% HCL at 200°F. for 30 seconds
(c) Sandblasted with #80 silica sand following the nitric acid etch and then activated with 10% HCL at the normal time and temperature of 5 seconds at 75°F.

TABLE XII

30.0% TEFLON 41BX
  3.0% ∅ octanoic acid (based on the TFE weight)
 10.0% alumina modified colloidal silica solids (based on the TFE weight)
  pH = 4.0   Conductivity = 1,720/70°F. micromhos

No.	Etch	V	A	CD	T	Thickness mil. -Calc.	Cracks(24X)	Appearance	Sintering Schedule	Adhesion
80	Yes	34	.5	19.2	2.0	1.29	Yes(100%)	Excellent	85'/750°	Good
81	Yes	34	.5	19.2	2.0	1.30	Yes(100%)	Excellent	10'/750°	Poor
82	Yes	34	.5	19.2	2.0	1.23	Yes(100%)	Excellent	90'/750°	Good*
83	Yes	34	.5	19.2	1.5	0.93	Yes(70%)	Excellent	120'/750°	Good*
84	Yes	34	.5	19.2	1.6	1.02	Yes(90%)	Excellent	60'/750°	Good*
86	Yes	34	.5	19.2	1.6	0.93	Yes(80%)	Excellent	30'/750°	Good*
87	Yes	34	.5	19.2	1.6	0.94	Yes(70%)	Excellent	4'/1000°	Good
88	Yes	34	.5	19.2	1.6	0.96	Yes(70%)	Excellent	4'/1000°	Good*
89	Yes	34	.5	19.2	1.6	0.94	Yes(60%)	Excellent	5'/1000°	Good
90	No	34	.5	19.2	1.6	0.38	Yes(20%)	Poor	5'/1000°	Good

* Panel sent to NRL for friction measurements.

8.

TABLE XIII

30.0% TEFLON 41BX
  3.0% ∅ octanoic acid (based on the TFE weight)
 20.0% alumina modified colloidal silica solids (based on the TFE Weight)
  pH = 4.0   Conductivity = 1,780/70°F. micromhos

No.	Etch	V	A	CD	T	Thickness mil. -calc.	Cracks(24X)	Appearance	Sintering Schedule	Adhesion
95	Yes	28	.5	19.2	1.6	0.92	Yes(100%)	Fair	10'/750°	Good
95	Yes	34	.5	19.2	1.6	0.86	Yes(100%)	Fair	10'/750°	Good
96	Yes	34	.5	19.2	1.0	0.65	Yes(60%)	Fair	10'/750°	Good
97	Yes	34	.5	19.2	0.8	0.52	Yes(40%)	Poor	10'/750°	Good
98	Yes	34	.5	19.2	0.6	0.40	Yes(40%)	Poor	10'/750°	Good

TABLE XIV

Bath formula: 30.0% TEFLON 41BX
  3.0% ∅ octanoic acid (based on the TFE weight)
 20.0% alumina modified colloidal silica solids (based on the TFE wt.)
 20.0% chrome oxide green pigment solids (based on the TFE weight)

No.	Etch	V	A	CD	T	Thickness mil.-calc.	Cracks(24X)	Appearance	Sintering Schedule	Adhesion
99	Yes	30	.5	19.2	1.6	0.98	Yes(90%)	Fair	10'/750°	Good
1	Yes	30	.5	19.2	1.4	0.64	Yes(60%)	Fair	10'/750°	Good
2	Yes	30	.5	19.2	1.4	0.66	Yes(50%)	Fair	10'/750°	Good
3	Yes	30	.5	19.2	1.2	0.56	Yes(40%)	Fair	10'/750°	Good
4	Yes	30	.5	19.2	1.0	0.40	Yes(10%)	Poor	10'/750°	Good
5	Yes	30	.5	19.2	1.0	0.43	Yes(10%)	Poor	10'/750°	Good

TABLE XV

Bath Formula:　30.0% TEFLON 41BX
3.0% ∅ octanoic acid (based on the TFE weight)
10.0% alumina modified colloidal silica solids (based on the TFE wt.)
20.0% chrome oxide green pigment solids (based on the TFE wt.)
pH = 4.0　　　　Conductivity = 2,050/70°F. micromhos

No.	Etch	V	A	CD	T	Thickness mil-calc.	Cracks	Appearance	Initial Sintering Cycle	Add'tl. Sintering Cycle	Adhesion
55	Yes	30	.5	19.2	1.0	0.59	No	Excellent	10'/700°	--	Fair [a]
56	Yes	30	.5	19.2	2.0	1.04	No	Excellent	10'/700°	--	Poor
57	Yes	30	.5	19.2	2.0	1.01	No	Excellent	10'/700°	90'/750°	Good*
58	Yes	30	.5	19.2	2.0	0.99	No	Excellent	10'/700°	90'/750°	Good*
59	Yes	30	.5	19.2	2.0	1.02	No	Excellent	10'/700°	--	Poor
60	Yes	30	.5	19.2	2.0	1.00	No	Excellent	10'/700°	15'/750°	Fair
61	Yes	30	.5	19.2	2.0	0.98	No	Excellent	10'/700°	30'/750°	Fair
62	Yes	30	.5	19.2	2.0	1.01	No	Excellent	10'/700°	30'/750°	Fair
63	Yes	30	.5	19.2	2.0	0.98	No	Excellent	10'/750°	120'/750°	Good
64	Yes	30	.5	19.2	2.0	0.97	No	Excellent	10'/750°	120'/750°	Good*
65	Yes	32	.5	19.2	2.0	1.00	No	Excellent	10'/800°	--	Fair
66	Yes	32	.5	19.2	2.0	1.02	No	Excellent	10'/800°	--	Fair
67	No	32	.5	19.2	2.0	0.65	Yes(10%)	Poor	10'/750°	--	Good [a]
68	Yes	32	.5	19.2	2.0	1.01	Yes(5%)	Fair	10'/750°	--	Poor
69	Yes	32	.5	19.2	2.0	0.97	No	Excellent	10'/750°	--	Poor
70	Yes	32	.5	19.2	2.0	0.99	No	Excellent	120'/750°	--	Good
71	Yes	30	.5	19.2	2.0	1.02	No	Excellent	60'/750°	--	Good
72	Yes	30	.5	19.2	2.0	1.00	No	Excellent	60'/750°	--	Good
73	Yes	30	.5	19.2	2.0	1.01	No	Good	30'/750°	--	Good*
74	Yes	30	.5	19.2	2.0	0.98	No	Excellent	120'/750°	--	Good
75	Yes	30	.5	19.2	2.0	0.97	No	Good	90'/750°	--	Good
76	Yes	30	.5	19.2	2.0	1.01	No	Good	90'/750°	--	Good
77	Yes	30	.5	19.2	2.0	1.00	No	Excellent	30'/750°	--	Good
78	Yes	30	.5	19.2	2.0	0.98	No	Excellent	20'/750°	--	Fair
79	Yes	30	.5	19.2	2.0	0.99	No	Excellent	20'/750°	--	Fair*

*Panel sent to NRL for friction measurements.
[a] Thin coating.

TABLE XVI

Bath Formula:　30.0% TEFLON 41BX
3.0% ∅ octanoic acid (based on the TFE weight)
pH = 4.0　　　　Conductivity = 1,550/70°F. micromhos

No.	Etch	V	A	CD	T	Thickness mil-calc.	Cracks	Appearance	Sintering Cycle	Adhesion
91	Yes	36	.5	19.2	1.6	1.14	Yes(5%)	Excellent	60'/750°	Poor-Fair
92	Yes	36	.5	19.2	1.6	1.05	No	Excellent	120'/750°	Good*
93	Yes	36	.5	19.2	1.6	1.06	No	Excellent	90'/750°	Fair [a]
94	Yes	36	.5	19.2	1.6	1.10	No	Excellent	10'/750°	Poor

* Panel sent to NRL for friction measurements.
[a] Adhesion of a narrow strip in the middle of the panel was "Good".

TABLE XVII

BOWDEN-LEBEN TESTS OF ELECTRODEPOSITED TFE COATINGS

Conditions: 10,000 Gm, Load — 100 Passes — Room Temperature — 0.1 cm/sec

Panel No.	Static Friction	Kinetic Friction (Average)	Final Kinetic Friction	Calculated Thickness Mils	Measured Thickness Mils	Comments
82	.08	.06	.09	1.23	1.25	Mudcracked - soft - too thick
88	.06	.07	.09	.96	1.60	High adhesion + cohesion - fine grained
84	.09	.09	.10	1.02	1.25	Low cohesion + adhesion - mud cracked
86	.06	.06	.08	.93	1.20	High cohesion + adhesion not as good as #88
83	.08	.09	.10	.93	1.25	Superficial extrusion — good cohesion and adhesion
79	.07	.07	.08	.99	1.75	Good adhesion --doughy cohesion - too thick
73	.08	.07	.07	1.01	1.20	Best of this formulation
57	.08	.07	.07	1.01	1.50	Too thick - extruded
64	.08	.09	.08	.97	1.50	Too thick - extruded
58	.05	.09	.09	.99	1.30	Second best of this formulation
92	.06	.07	.08	1.05	1.25	Best of panels without pigment

TABLE XVII

TABLE XVIII

Effect of Sintering Time and Temperature on the Adhesion of Electrodeposited TFE Films to Steel Substrates.

Bath Formula: 30.0% TEFLON 41BX
3.0% ∅ octanoic acid (based on the TFE weight)
10.0% alumina modified colloidal silica solids (based on the TFE wt.)
20.0% chrome oxide green pigment solids (based on the TFE wt.)
pH = 4.0   Conductivity = 2,050/70°F. micromhos

Substrate - 1020 cold rolled steel
Surface Preparation - sandblasted with #80 silica sand.
Etch - 5-6 seconds at room temperature.

No.	Etch	V	A	CD	T	Thickness mil-calc	Cracks	Appearance	Sintering Time (min)	Oven Temp. °F	Peak Surface Temp °F.	Adhesion Rating (a)
20	Yes	30	.5	19.2	2.0	1.19	No	Excellent	15'	750°F.	770°F.	1
21	Yes	30	.5	19.2	2.0	1.10	No	Excellent	30'	750°F.	770°F.	3
22	Yes	30	.5	19.2	2.0	1.10	No	Excellent	60'	750°F.	770°F.	7
23	Yes	30	.5	19.2	2.0	1.09	No	Excellent	120'	750°F.	770°F.	8
24	Yes	30	.5	19.2	2.0	1.14	No	Excellent	10'	700°F.	720°F.	1
25	Yes	35	.5	19.2	1.8	1.11	No	Excellent	90'	700°F.	720°F.	3
26	Yes	35	.5	19.2	1.8	1.09	No	Excellent	60'	700°F.	720°F.	2
27	Yes	32	.5	19.2	1.7	1.01	No	Excellent	30'	700°F.	720°F.	1
28	Yes	32	.5	19.2	1.7	1.06	No	Excellent	120'	700°F.	720°F.	5
29	Yes	32	.5	19.2	1.7	1.01	No	Excellent	90'	750°F.	770°F.	8
30	Yes	32	.5	19.2	1.7	1.08	No	Excellent	10'	640°F.		1
31	Yes	32	.5	19.2	1.7	1.02	No	Excellent	10'	615°F.		1
32	Yes	32	.5	19.2	1.7	1.06	No	Excellent	10'	612°F.		1
33	Yes	32	.5	19.2	1.7	0.99	No	Excellent	120'	612°F.		3
34	Yes	32	.5	19.2	1.7	1.06	No	Excellent	90'	612°F.		2
35	Yes	32	.5	19.2	1.7	1.06	No	Excellent	60'	612°F.		2
36	Yes	32	.5	19.2	1.7	1.02	No	Excellent	10'	1000°F.	930°F.*	10
37	Yes	32	.5	19.2	1.7	1.00	No	Excellent	7'	1000°F.		8
38	Yes	32	.5	19.2	1.7	0.97	No	Excellent	7'	1000°F.*	890°F*	7
39	Yes	32	.5	19.2	1.7	1.03	No	Excellent	6'	1000°F*	865°F*	4
40	Yes	32	.5	19.2	1.7	1.00	No	Excellent	5'	1000°F*	800°F*	2
41	Yes	32	.5	19.2	1.7	0.96	No	Excellent	6.5'	1000°F*	875°F*	6
42	Yes	32	.5	19.2	1.7	0.98	No	Excellent	10'	900°F*	870°F*	9
43	Yes	32	.5	19.2	1.7	0.95	No	Excellent	8'	900°F*	845°F*	4
44	Yes	32	.5	19.2	1.7	1.08	No	Excellent	9'	900°F*	862°F*	7
45	Yes	32	.5	19.2	1.7	1.11	No	Excellent	14'	850°F*	862°F*	7
46	Yes	32	.5	19.2	1.7	1.04	No	Excellent	10'	850°F*	830°F*	4
47	Yes	32	.5	19.2	1.7	1.05	No	Excellent	16'	800°F*	815°F*	6
48	Yes	32	.5	19.2	1.7	1.09	No	Excellent	20'	800°F*	820°F*	7
49	Yes	32	.5	19.2	1.7	1.01	No	Excellent	20'	800°F*	823°F*	6 (b)
50	Yes	32	.5	19.2	1.7	1.00	No	Excellent	22'	800°F*	823°F*	3 (c)
52	Yes	32	.5	19.2	1.7	0.97	No	Excellent	14'	850°F		7
53	Yes	32	.5	19.2	1.7	0.97	No	Excellent	14'	850°F		1 (c)

*See Table XIX

(a) Adhesion Ratings - A 5 rating is ideal
    Reading toward 1 indicates lessening peel strength
    Reading toward 10 indicates increasing softness
    and degradation of the film

(b) Cooled in front of fan.
(c) Quenched immediately in water.

49.

TABLE XIX
Panel Surface Temperatures (°F.)

Elapsed Time (min.)	Oven Temp. 1000°F. #36	#38	#39	#40	#41	Oven Temp. 900°F. #42	#43	#44	Oven Temp. 850°F. #45	#46	Oven Temp. 800°F. #47	#48	#49	#50	Oven Temp. 750°F. #29	Oven Temp. 700°F. #28
0	80	80	100	80	100	100	100	80	80	100	80	80	80	80	80	80
0.5	250	320	320	240	260	240	260	245	250	210	240	235	220	210	240	280
1.0	396	390	410	340	360	325	355	320	320	280	315	300	270	260	290	345
1.5	460	467	480	420	–	390	430	405	400	350	370	360	350	340	340	370
2.0	540	530	550	490	500	460	–	460	450	415	425	405	405	400	390	410
2.5	610	600	610	565	580	530	–	530	500	460	470	470	455	450	435	450
3.0	660	650	670	625	630	580	590	580	550	520	515	520	505	502	470	480
3.5	710	700	715	670	680	630	630	625	590	560	560	560	540	540	500	505
4.0	750	740	760	720	730	670	670	660	630	605	590	590	580	575	525	520
4.5	790	770	790	760	770	705	700	700	660	640	620	620	615	620	545	550
5.0	810	810	820	<u>800</u>	800	735	730	730	690	670	650	650	645	645	570	570
5.5	840	835	845	730	830	760	–	760	720	705	670	670	670	670	590	580
6.0	870	860	<u>865</u>	700	860	780	790	780	740	725	695	695	690	695	610	590
6.5	880	880	–	655	<u>875</u>	800	805	800	760	745	715	720	710	715	625	600
7.0	890	<u>890</u>	720	620	820	815	820	815	775	765	730	730	730	730	655	625
7.5	895	825	710	590	760	830	832	830	790	780	740	740	740	745	680	650
8.0	900	755	665	550	710	840	<u>845</u>	842	800	790	755	755	750	755	–**	–**
8.5	907	695	–	520	660	850	–	855	810	805	765	765	760	765	–	–
9.0	915	650	600	495	620	860	720	<u>862</u>	820	810	770	770	770	770	–	–
9.5	922	610	–	465	590	865	–	795	830	820	775	780	780	780	710	685
10.0	<u>930</u>	565	530	450	550	<u>870</u>	620	735	835	<u>830</u>	785	787	785	785	720	–
10.5	C	530	–	–	–	800	–	675	840	–	790	790	790	790	710	720
11.0	O	500	465	400	–	750	550	630	845	715	795	795	795	795	730	720
11.5	O	475	–	–	450	690	–	590	850	–	798	800	800	800	735	720
12.0	L	444	415			650	490	560	852	640	800	802	802	803	745	720
12.5	I	420				610		520	855	610	805	803	805	805	770	720
13.0	N	400	380			580	440	490	858	570	807	805	807	807	760	720
13.5	G	380			370	545		470	860		808	808	810	809	–	–
14.0		360	345			510	400	440	<u>862</u>	510	810	810	812	811	770	720
14.5		340				480	–		815		811	810	813	812		
15.0		325	310			450	360		740	450	812	812	815	813		
15.5		310		360					690		813	813	817	814		
16.0		300							640		<u>815</u>	815	818	815		
16.5		285							600			816	819	817		
17.0		275							565	370	700	817	820	818		
17.5		260								340	670	818	820	879		
18.0		250		220			280		510		620	819	820	820		
18.5		243							470		580	819	821	821		
19.0		235							440		545	819	821	822		
19.5		230									515	820	822	823		
20.0		220		200			280				480	<u>820</u>	823	<u>823</u>		
20.5		210									460	735	690	823		
21.0											440	680	585	823		
21.5											420	640	520	823		
22.0											400	610	450	<u>823</u>		
22.5												560	400	200		
23.0												525	350	130		
23.5												500	315	80		

** L & N "Speedomax" used—erratic above this point—indicated temps.

Note: Where underlined, panel was removed from oven. Example: <u>823</u>

50.

TABLE XX

Bath Formula: 30.0% TEFLON 41BX

3.0% ∅ octanoic acid (based on the TFE weight)

pH - 4.0   Conductivity = 1,290/70°F. micromhos

Substrate-SAE 1010
cold rolled steel
Surface Preparation-
sandblasted with #80
silica sand
Etch - 5-6 seconds at
room temperature

No.	Etch	V	A	CD	T	Thick mil calc	Sintering Schedule	On edge cracks	crazing	In Interior cracks	crazing	% wt. loss	Appearance (naked eye)	Adhesion Rating
111	Yes	48	.45	17.3	1.7	1.17	7.0/800°F.	Rdm	Lt	No	Hvy	7.3	Ex	1
112	Yes	48	.45	17.3	1.5	1.05	9.0/800°F.	Rdm	Rdm	No	Hvy	4.5	Ex	1
113	Yes	48	.45	17.3	1.2	.90	11.0/800°F.	Rdm	Lt	No	Hvy	5.2	Ex	3
114	Yes	48	.48	18.4	1.0	.79	14.5/800°F.	Rdm	Lt	Rdm	Hvy	6.9	Ex	4
115	Yes	48	.48	18.4	.8	.62	20.0/800°F.	No	Lt	No	Rdm	10.1	Ex	6
116	Yes	48	.49	18.8	.8	.70	12.0/800°F.	No	Rdm	No	Lt	4.5	Ex	3
117	Yes	48	.50	19.2	.8	.29	14.0/800°F.	No	Rdm	No	No	6.8	Fair	4
118	Yes	48	.51	19.6	.8	.71	16.0/800°F.	No	Lt	No	Med	5.5	Ex	5
119	Yes	48	.52	20.0	.8	.65	18.0/800°F.	No	Lt	No	Lt	7.8	Ex	5
120	Yes	48	.51	19.6	.8	.71	20.0/800°F.	No	Lt	No	Med	7.7	Ex	6
121	Yes	48	.49	18.8	.8	.63	25.0/800°F.	No	Med	No	Lt	13.0	Ex	7
122	Yes	48	.49	18.8	.8	.70	30.0/800°F.	No	Lt	No	Med	14.4	Ex	9
123	Yes	48	.49	18.8	.8	.70	20.0/800°F.	No	Lt	No	Lt	11.2	Ex	6
124	Yes	48	.49	18.8	.8	.66	20.0/800°F.	No	Lt	No	Lt	9.5	Ex	6
125	Yes	48	.49	18.8	.8	.70	20.0/800°F.	No	Lt	No	Med	9.0	Ex	6
126	Yes	48	.53	20.2	.8	.84	22.5/800°F.	No	Lt	No	Med	10.7	Ex	7
127	Yes	36	.50	19.2	.8	.75	22.5/800°F.	No	Lt	No	Med	12.5	Ex	7
128	Yes	36	.49	18.8	.8	.71	6.0/800°F.	No	Lt	No	Lt	3.9	Ex	1
129	Yes	36	.49	18.8	.6	.50	20.0/800°F.	No	Rdm	No	Lt	16.0	Good	6
130	Yes	36	.49	18.8	.6	.41	6.0/800°F.	No	Rdm	No	Lt	4.7	Good	1

Evidence of cracking & crazing (Viewed under 24X)

Fan Dried - Yes
Oven Dried (30'/180°F.) - No
Water Quenched - No

TABLE XXI

Bath Formula: 30.0% TEFLON 41BX
3.0% ∅ octanoic acid (based on the TFE weight)
0.5% carbon black solids (based on the TFE weight)

Substrate-SAE 1010 cold rolled steel
Surface Preparation – sandblasted with #80 silica sand
Etch –5–6 seconds at room temperature
Fan Dried – Yes
Oven Dried (30'/180°F.)-No
Water Quenched-No

No.	Etch	V	A	CD	T	mil calc	Sintering Schedule	On edge cracks	crazing	In Interior cracks	crazing	% wt. loss	Naked Eye	Adhesion Rating
131	Yes	36	.65	25.0	.6	.66	6.0'/800°F.	No	Rdm	No	Lt	3.6	Good	4
132	Yes	36	.55	21.2	.6	.55	6.0'/800°F.	No	Rdm	No	Rdm	4.2	Fair	3
133	Yes	32	.50	19.2	.6	.48	20.0'/800°F.	No	Rdm	No	Rdm	15.3	Poor	6

Bath Formula: 30.0% TEFLON 41BX
3.0% ∅ octanoic acid (based on the TFE weight)
1.0% carbon black solids (based on the TFE wt.)

No.	Etch	V	A	CD	T	mil calc	Sintering Schedule	On edge cracks	crazing	In Interior cracks	crazing	% wt. loss	Naked Eye	Adhesion Rating
134	Yes	32	.50	19.2	.6	.49	6.0'/800°F.	No	No	No	No	4.8	Fair	3
135	Yes	32	.50	19.2	.6	.42	20.0'/800°F.	No	Rdm	No	No	13.0	Poor	7
136	Yes	32	.52	20.0	.8	.84	20.0'/800°F.	No	Lt	No	Lt	11.1	Good	5

Bath Formula: 30.0% TEFLON 41BX
3.0% ∅ octanoic acid (based on the TFE weight)
2.0% carbon black solids (based on the TFE wt.)

No.	Etch	V	A	CD	T	mil calc	Sintering Schedule	On edge cracks	crazing	In Interior cracks	crazing	% wt. loss	Naked Eye	Adhesion Rating
137	Yes	20	.27	10.4	.8	.48	20.0'/800°F.	No	Rdm	No	No	16.1	Poor	7
138	Yes	20	.30	11.5	1.0	.57	20.0'/800°F.	No	Lt	No	Rdm	11.0	Poor	7
139	Yes	20	.30	11.5	1.0	.45	20.0'/800°F.	No	Rdm	No	No	17.2	Poor	7
140	Yes	20	.30	11.5	1.2	.57	20.0'/800°F.	No	Lt	No	Rdm	16.7	Poor	7
141	Yes	20	.30	11.5	1.4	.61	12.0'/800°F.	No	Lt	No	Lt	7.6	Good	3
142	Yes	20	.28	10.8	1.8	.70	16.0'/800°F.	No	Med	No	Lt	14.0	Good	5
143	Yes	20	.28	10.8	2.5	.84	20.0'/800°F.	No	Med	No	Med	11.6	Good	6
144	Yes	20	.28	10.8	3.0	1.02	20.0'/800°F.	No	Rdm	No	Lt	3.8	Ex	1

Bath Formula: 30.0% TEFLON 41BX
3.5% ∅ octanoic acid (based on the TFE weight)
3.0% carbon black solids (based on the TFE wt.)

No.	Etch	V	A	CD	T	mil calc	Sintering Schedule	On edge cracks	crazing	In Interior cracks	crazing	% wt. loss	Naked Eye	Adhesion Rating
145	Yes	36	.58	22.5	.7	.61	7.0'/800°F.	No	Rdm	No	Rdm	3.9	Good	1
146	Yes	32	.40	15.4	.7	.44	7.0'/800°F.	No	Rdm	No	Rdm	3.6	Poor	1
147	Yes	40	.60	23.0	1.4	.86	7.0'/800°F.	No	Rdm	No	Rdm	2.7	Ex	1
148	Yes	34	.45	17.3	.7	.66	18.0'/800°F.	No	Lt	No	Lt	14.1	Good	6
149	Yes	36	.50	19.2	.7	.66	18.0'/800°F.	No	Lt	No	Rdm	15.4	Good	6
150	Yes	36	.50	19.2	.7	.68	15.0'/750°F.	No	Rdm	No	Rdm	10.3	Good	2
151	Yes	36	.50	19.2	.7	.66	7.0'/800°F.	No	Rdm	No	Rdm	3.6	Good	1
152	Yes	36	.50	19.2	.7	.60	12.0'/800°F.	No	Lt	No	Rdm	6.5	Good	2
153	Yes	36	.50	19.2	.7	.58	13.0'/800°F.	No	Lt	No	Rdm	10.1	Good	2
154	Yes	36	.50	19.2	.7	.49	14.0'/800°F.	No	Lt	No	Rdm	11.1	Fair	2
155	Yes	36	.50	19.2	.7	.57	15.0'/800°F.	No	Lt	No	Rdm	11.1	Fair	4

TABLE XXII

Bath Formula: 30.0% TEFLON 41BX
3.0% ∅ octanoic acid (based on the TFE weight)
2.0% carbon black solids (based on the TFE wt.)
pH = 4.0

Substrate-SAE 1010
cold rolled steel
Surface Preparation –
sandblasted with #80
silica sand
Etch – 5-6 seconds at
room temperature

						Thick mil calc.	Sintering Schedule	Quench	Evidence of cracking & crazing (Viewed under 24X)				%wt. loss	Appearance (naked eye)
									On edge		In interior			
No.	Etch	V	A	CD	T				crack	crazing	crack	crazing		
157	Yes	30	.48	18.4	.7	.44	16.0/800°F.	No	No	Lt	No	No	14.3	Poor
159	Yes	30	.48	18.4	.7	.44	16.0/800°F.	No	No	Lt	No	Rdm	13.6	Fair
161	Yes	36	.52	19.9	.7	.59	18.0/800°F.	No	No	Lt	No	Lt	13.1	Fair
160	Yes	36	.52	19.9	.7	.59	20.0/800°F.	No	No	Lt	No	Lt	13.4	Good
162	Yes	36	.52	19.9	1.0	.77	20.0/800°F.	No	No	Med	No	Med	12.9	Good
166	Yes	36	.52	19.9	1.0	.82	25.0/775°F.	No	No	Lt	No	Lt	-	Good
167	Yes	38	.50	19.2	1.0	.61	90.0/700°F.	No	No	Lt	No	Rdm	11.5	Good
168	Yes	38	.40	15.4	1.0	.63	90.0/700°F.	No	No	Lt	No	Lt	12.5	Good
178	Yes	42	.54	20.7	1.0	.78	45.0/750°F.	No	No	Med	No	Lt	12.0	Ex
179	Yes	40	.50	19.2	1.0	.73	45.0/750°F.	No	No	Med	No	Lt	-	Good
180	Yes	40	.58	22.2	.8	.76	45.0/750°F.	No	No	Med	No	Li	-	Good
181	Yes	40	.64	24.6	.9	.87	30.0/750°F.	No	No	Med	No	Lt	5.3	Ex
182	Yes	40	.62	23.8	.6	.59	15.0/750°F.	No	No	Rdm	No	Rdm	5.5	Good
183	Yes	50	.78	29.9	.4	.57	15.0/750°F.	No	No	Rdm	No	Rdm	--	Good
184	Yes	50	.90	34.5	.4	.61	45.0/750°F.	No	No	Lt	No	Rdm	--	Good
185	Yes	50	.90	34.5	.4	.63	30.0/750°F.	No	No	Med	No	Lt	-	Good
186	Yes	50	.86	33.0	.3	.56	30.0/750°F.	No	No	Lt	No	Rdm	-	Good
187	Yes	50	.80	30.7	.2	.43	20.0/750°F.	No	No	Lt	No	Rdm	-	Fair
188	Yes	50	.80	30.7	.2	.36	45.0/750°F.	No	No	No	No	No	-	Poor
189	Yes	50	.80	30.7	.2	.42	45.0/750°F.	No	No	Rdm	No	No	-	Poor

Bath Formula: 30.0% TEFLON 41BX
3.0% ∅ octanoic acid (based on the TFE weight)
10.0% alumina modified colloidal silica solids
(based on the TFE weight)
2.0% carbon black solids (based on the TFE wt.)
pH = 4.0

No.	Etch	V	A	CD	T	Thick mil calc.	Sintering Schedule	Quench	crack	crazing	crack	crazing	%wt. loss	Appearance
163	Yes	34	.50	19.2	1.0	.69	90.0/700°F.	No	No	Med	No	Lt	11.4	Good
164	Yes	34	.50	19.2	1.6	.41	90.0/700°F.	No	No	Lt	No	Rdm	13.5	Fair
165	Yes	34	.50	19.2	.6	.54	90.0/700°F.	No	No	Med	No	Lt	11.6	Fair

Pan Dried – Yes
·Oven Dried (30'/180°F.) – No
Pigment flocculation – None
      (24X)

53.

TABLE XXIII

Substrate-SAE 1010
  cold rolled steel
Surface Preparation –
  sandblasted with #80
  silica sand
Etch – 5-6 seconds at
  room temperature

Bath Formula: 30.0% TEFLON 41BX
              3.0% ∅ octanoic acid (based on the TFE weight)
              10.0% carbon black solids (based on the TFE wt.)
              pH = 4.0

No.	Etch	V	A	CD	T	Thick mil calc.	Sintering Schedule	Quench	Evidence of cracking & crazing (Viewed under 24X)				Appearance Naked eye
									On edge		In Interior		
									crack	crazing	crack	crazing	
190	Yes	40	.99	38.0	.8	.47	120.0/700°F.	No	No	Lt	No	Rdm	Fair
191	Yes	40	.95	36.5	.8	.43	120.0/700°F.	No	No	Med	No	Rdm	Fair
192	Yes	30	.70	26.8	.8	.24	120.0/700°F.	No	No	Lt	No	Rdm	Poor
193	Yes	30	.70	26.9	.8	.29	120.0/700 F.	No	No	Lt	No	Rdm	Poor
194	Yes	20	.44	16.9	3.0	.87	120.0/700°F.	No	Rdm	Med	Lt	Lt	Good
195	Yes	24	.50	19.2	3.0	1.22	15.0/700°F.	No	Lt	Med	Lt	Med	Ex

Bath Formula: 30.0% TEFLON 41BX
              4.0% ∅ octanoic acid (based on the TFE weight)
              10.0% carbon black solids (based on the TFE wt.)

No.	Etch	V	A	CD	T	Thick mil calc.	Sintering Schedule	Quench	crack	crazing	crack	crazing	Appearance
196	Yes	30	.65	24.9	3.0	1.37	15.0/700°F.	No	Rdm	Rdm	Rdm	Rdm	Ex

Bath Formula 30.0% TEFLON 41BX

pH = 4.0   5.0% ∅ octanoic acid (based on the TFE weight)
Conductivity = 2,240/70°F.   10.0% carbon black solids (based on the TFE wt.)
             micromhos

No.	Etch	V	A	CD	T	Thick mil calc.	Sintering Schedule	Quench	crack	crazing	crack	crazing	Appearance	
197	Yes	24	.50	19.2	2.0	1.30	15.0/700°F.	No	Rdm	Hvy	Rdm	Hvy	Ex	a
198	Yes	24	.50	19.2	1.0	.77	15.0/700°F.	No	Rdm	Med	No	Lt	Good	a
199	Yes	24	.50	19.2	1.0	.78	120.0/700°F.	No	Rdm	Med	No	Lt	Good	a
200	Yes	24	.50	19.2	.8	.68	120.0/700°F.	No	Rdm	Med	No	Lt	Good	a
201	Yes	24	.50	19.2	.8	.63	120.0/700°F.	No	Rdm	Med	No	Lt	Fair	b
202	Yes	24	.50	19.2	.5	.48	90.0/700°F.	No	No	Lt	No	No	Fair	c
203	Yes	40	.70	26.9	.2	.54	90.0/700°F.	No	No	Lt	No	No	Good	··

a = Rebake 2 hours at 700°F.
b = Cooled down in oven to below 620°F. by shutting oven off.
c = Cooled down to room temperature in oven overnight.

Fan Dried – Yes
Oven Dried (30'/180°F.) – No
Water Quenched – No
Pigment flocculation (24X) – No

TABLE XXIII - continued

Bath Formula: 30.0% TEFLON 41BX
3.0% ø octanoic acid (based on the TFE weight)
3.0% lampblack solids
20.0% FEP solids (based on the TFE weight)

No.	Etch	V	A	CD	T	Thick mil calc.	Sintering Schedule	Quench	On edge crack	On edge crazing	In Interior crack	In Interior crazing	Appearance naked eye
169	Yes	32	.30	11.5	2.0	.74	120.0/700°F.	No	No	Lt	No	No	Good
170	Yes	36	.40	15.4	2.0	.91	120.0/700°F.	No	No	Lt	No	No	Ex
171	Yes	44	.60	23.0	1.0	.78	120.0/700°F.	No	Rdm	Rdm	No	No	Good
172	Yes	40	.50	19.2	.8	.50	90.0/700°F.	No	No	Rdm	No	No	Fair
173	Yes	40	.50	19.2	1.0	.59	45.0/750°F.	No	No	Rdm	No	No	Fair
174	Yes	36	.40	15.4	1.0	.50	45.0/750°F.	No	No	Rdm	No	No	Fair
175	Yes	42	.50	19.2	1.0	.70	45.0/750°F.	No	Rdm	Rdm	No	No	Good
176	Yes	42	.50	19.2	1.0	.68	120.0/700°F.	No	Rdm	Rdm	No	No	Good
177	Yes	42	.35	13.4	.8	.31	90.0/700°F.	No	No	No	No	No	Poor

Fan Dried - Yes
Oven Dried (30'/180°F.) - No
Water Quenched - No
Pigment flocculation (24X) - No.

TABLE XXIV

Bath Formula: 30.0% TEFLON 41BX

Substrate-SAE 1010 cold rolled steel

Surface Preparation- sandblasted with #80 silica sand

Etch - 5-6 seconds at room temperature

3.0% ∅ octanoic acid (based on the TFE weight)

10.0% alumina modified colloidal silica solids (based on the TFE weight)

10.0% FEP solids (based on the TFE weight)

pH ≈ 4.5   Conductivity = 1,520/70°F. micromhos

						Thick mil calc	Fan Dried	Oven Dried	Sintering Schedule	Quench	Evidence of cracking & crazing (Viewed under 24X)			
											On Edge		In Interior	
No.	Etch	V	A	CD	T						cracks	crazing	cracks	crazing
207	Yes	38	.50	19.2	2.0	1.23	Yes	Yes	45'/750°F.	No	Hvy	v.Hvy	Lt	v.Hvy
208	Yes	38	.50	19.2	1.0	.68	Yes	Yes	45'/750°F.	No	Hvy	v.Hvy	Lt	Med
209	Yes	38	.50	19.2	1.0	.64	Yes	Yes	45'/750°F.	No	Hvy	v.Hvy	Lt	Med.
210	Yes	38	.50	19.2	1.0	.55	Yes	Yes	30'/750°F.	No	Hvy	v.Hvy	Rdm	Lt
211	Yes	38	.50	19.2	1.0	.64	Yes	Yes	30'/750°F.	No	Hvy	v.Hvy	Rdm	Med
212	Yes	38	.50	19.2	1.0	.70	Yes	Yes	30'/750°F.	No	Hvy	v.Hvy	Lt	Hvy
213	Yes	38	.50	19.2	1.0	.67	Yes	Yes	30'/750°F.	No	Hvy	v.Hvy	Lt	Hvy
214	Yes	38	.50	19.2	1.0	.65	Yes	Yes*	30'/750°F.	No	Hvy	v.Hvy	Hvy	Hvy
215	Yes	38	.50	19.2	1.0	.66	Yes	Yes*	40'/750°F.	No	Hvy	v.Hvy	Med	v.Hvy
216	Yes	38	.50	19.2	1.0	.67	No	Yes*	40'/750°F.	No	Hvy	v.Hvy	Lt	Hvy

General Appearance (naked eye) - Good, although all samples exhibit various degrees of staining, due to the liberation of Fe^{+++} from the substrate.

Bath Formula: 30.0% TEFLON 41BX

3.0% ∅ octanoic acid (based on the TFE weight)

10.0% alumina modified colloidal silica solids (based on the TFE wt)

10.0% FEP solids (based on the TFE weight)

10.0% chrome oxide green pigment solids (based on the TFE wt.)

pH = >4.5   Conductivity = 1,850/70°F. micromhos

217	Yes	38	.60	23.0	1.0	.70	Yes	Yes**	30'/750°F.	No	Med	Hvy	Rdm	Lt
218	Yes	38	.60	23.0	1.0	.70	Yes	No	30'/750°F.	No	Med	Hvy	Rdm	Lt
219	Yes	60	1.10	28.8	.5	.75	Yes	Yes	30'/750°F.	No	Lt	Med	No	Lt
220	Yes	100	2.00	52.2	.2	.67	Yes	No	30'/750°F.	No	Hvy	Lt	No	Lt
222	Yes	100	2.00	52.2	.2	.65	Yes	No	30'/750°F.	No	Lt	Hvy	Rdm	Med
223	Yes	10	.15	3.9	4.0	.63	Yes	No	30'/750°F.	No	Hvy	Lt	No	Med
224	Yes	10	.15	3.9	6.0	.82	Yes	No	15'/750°F.	No	No	Lt	No	Rdm
225	Yes	10	.15	3.9	4.0	.52	Yes	No	15'/750°F.	No	No	Lt	No	No
226	Yes	10	.15	3.9	4.0	.55	Yes	No	15'/750°F.	No	No	Lt	No	No

*Oven dried - 15 minutes at 180°F.

** Oven dried - 5 minutes at 180°F.

Oven Dried = 30' at 180°F. (unless otherwise noted)

General Appearance - Good (above thickness 0.8 mil) Poor to fair (below a thickness of 0.8 mil)

Color - Even medium green.

Pigment flocculation (24X) - Little or none.

TABLE XXV

Substrate-SAE 1010
cold rolled steel
Surface Preparation -
sandblasted with #80
silica sand.
Etch - 5-6 seconds at
room temperature

Bath Formula: 30.0% TEFLON 41BX
3.0% ∅ octanoic acid (based on the TFE weight)
10.0% alumina modified colloidal silica solids (based on the TFE weight)
10.0% FEP solids (based on the TFE weight)
20.0% chrome oxide green pigment solids (based on the TFE weight)

No	Etch	V	A	CD	T	Thick mil calc	Fan Dried	Oven Dried	Sintering Schedule	Quench	On Edge cracks	crazing	In Interior cracks	crazing
231	Yes	38	.72	27.6	1.0	.79	Yes	No	10'/750°F.	No	Lt	Hvy	No	Rdm
227	Yes	28	.52	19.9	0.5	.43	Yes	No	30'/750°F.	No	Rdm	Med	No	Rdm
228	Yes	36	.60	23.0	3.0	1.06	Yes	Yes	30'/750°F.	Yes	Rdm	Rdm	No	Rdm
229	Yes	28	.70	26.8	2.0	.63	Yes	No	30'/750°F.	Yes	No	No	No	No
230	Yes	28	.60	23.0	2.5	.82	Yes	No	30'/750°F.	Yes	Lt	Rdm	Rdm	Lt
232	Yes	30	.60	23.0	2.5	.79	Yes	Yes	30'/750°F.	No	Lt	Hvy	Rdm	Lt
233	Yes	30	.60	23.0	2.5	.77	Yes	Yes	30'/750°F.	No	Rdm	Hvy	No	Rdm
234	Yes	30	.60	23.0	2.5	.81	Yes	Yes	15'/750°F.	No	Lt.	Hvy	No	Lt
235	Yes	30	.50	19.2	2.5	.66	Yes	Yes	15'/750°F.	No	Lt	Hvy	No	Lt
236	Yes	30	.64	24.5	2.5	.91	Yes	No	45'/750°F.	No	Lt	Hvy	No	Rdm
237	Yes	32	.66	25.3	4.0	1.42	Yes	Yes*	45'/750°F.	No	Hvy	Hvy	Lt	Med
238	Yes	32	.62	23.8	3.5	1.34	Yes	Yes*	30'/750°F.	Yes	No	Rdm	No	Rdm
239	Yes	32	.60	23.0	3.2	1.12	Yes	Yes*	15'/750°F.	No	Lt	Med	Rdm	Med
240	Yes	28	.60	23.0	3.5	1.11	Yes	Yes*	30'/750°F.	Yes	No	Rdm	No	Rdm
241	Yes	28	.66	25.3	3.7	1.33	Yes	Yes*	45'/750°F.	Yes	No	Rdm	No	Lt
242	Yes	28	.68	26.1	3.5	1.33	Yes	Yes*	30'/750°F.	No	Med	Hvy	Lt	Hvy
243	Yes	28	.60	23.0	3.5	1.22	Yes	Yes*	45'/750°F.	Yes	Rdm	Rdm	No	Rdm

Note: column header "Evidence of cracking & crazing (Viewed under 24X)" spans the last four columns (On Edge: cracks, crazing; In Interior: cracks, crazing).

* Oven dried 5 minutes at 180°F.
Oven Dried - 30' at 180°F. (unless otherwise noted)
General appearance (naked eye) - Excellent (except #227, #229, and #235 which show poor hiding in the middle areas due to a low average thickness.)
Color - Even medium green.
Pigment flocculation - (24X) - Little or none.

TABLE XXVI

Bath Formula: 30.0% TEFLON 41BX

Substrate-SAE 1010 cold rolled steel
Surface Preparation— Sandblasted with #80 silica sand
Etch – 5-6 Seconds at room temperature

3.0% ø octanoic acid (based on the TFE weight)
20.0% chrome oxide green pigment solids (based on the TFE weight)
pH = 4.0   Conductivity= 2,150/70° micromhos

No.	Etch	V	A	CD	T	Thick mil calc	Fan Dried	Oven Dried	Sintering Schedule	Quench	On Edge cracks	On Edge crazing	In Interior cracks	In Interior crazing
244	Yes	30	.55	21.2	2.0	.95	Yes	Yes	30/750°F.	No	No	No	Rdm	No
245	Yes	28	.50	19.2	2.0	.72	Yes	Yes	30/750°F.	No	No	No	No	No
246	Yes	28	.48	18.4	2.2	.77	Yes	Yes	30/750°F.	Yes	No	No	No	No
247	Yes	28	.48	18.4	2.3	.70	Yes	Yes	30/750°F.	Yes	No	No	No	No
248	Yes	28	.50	19.2	2.4	.88	Yes	Yes	45/750°F.	No	No	No	No	No
249	Yes	28	.50	19.2	2.4	.87	Yes	Yes	15/750°F.	No	No	No	No	No
250	Yes	28	.52	19.9	2.3	.94	Yes	No	30/750°F.	No	No	Rdm	No	Rdm
251	Yes	28	.52	19.9	2.1	.92	Yes	No	30/750°F.	No	No	Rdm	No	Rdm
252	Yes	28	.50	19.2	2.0	.82	Yes	No	30/750°F.	No	No	Rdm	No	Rdm
253	Yes	28	.48	19.2	2.0	.84	Yes	No	30/750°F.	Yes	No	No	No	No
254	Yes	28	.48	18.4	2.0	.84	Yes	No	30/750°F.	Yes	No	No	No	No
255	Yes	26	.50	19.2	2.0	.85	Yes	Yes	15/750°F.	Yes	No	No	No	No
256	Yes	26	.50	19.2	2.0	.77	Yes	Yes	45/750°F.	Yes	No	No	No	No
257	Yes	26	.49	19.2	3.0	.96	Yes	Yes	45/750°F.	No	No	No	No	No
258	Yes	26	.50	17.3	4.0	1.42	Yes	Yes	45/750°F.	No	No	No	No	No
259	Yes	26	.50	19.2	3.5	1.20	Yes	Yes	45/750°F.	No	No	No	No	No
260	Yes	26	.50	19.2	3.5	1.29	Yes	No	45/750°F.	Yes	No	No	No	No
261	Yes	26	.50	17.6	3.5	1.11	Yes	No	30/750°F.	No	No	No	No	No
262	Yes	26	.47	18.0	3.5	1.12	Yes	No	15/750°F.	No	No	No	No	No
263	Yes	28	.49	18.8	3.5	1.19	Yes	No	30/750°F.	Yes	No	No	No	No
264	Yes	28	.51	19.6	3.5	1.11	Yes	No	15/750°F.	Yes	No	No	No	No

Oven Dried – 30' at 180°F.
General Appearance (naked eye) – Excellent
Color – Even medium green
Pigment flocculation – (24X) – Slight (sometimes heavy near the edges)

TABLE XXVII

Bath Formula: 30.0% TEFLON 41 BX

Substrate-SAE 1010 cold rolled steel
Surface Preparation – sandblasted with #80 silica sand.
Etch – 5–6 seconds at room temperature.

3.0% ∅ octanoic acid (based on the TFE weight)
20.0% chrome oxide green pigment solids (based on the TFE weight)
20.0% FEP solids (based on the TFE weight)
pH => 4.0 Conductivity=1,900/70°F. micromhos

No.	Etch	V	A	CD	T	Thick mil calc.	Fan Dried	Oven Dried	Sintering Schedule	Quench	On Edge cracks	crazing	In Interior cracks	crazing
265	Yes	28	.5	19.2	2.5	.80	Yes	Yes	45'/750°F.	Yes	Hvy.	No	Lt.	No
266	Yes	28	.5	19.2	2.5	.73	Yes	Yes	45'/750°F.	No	v.Hvy	Med.	Med.	Med.
267	No	28	.5	19.2	2.5	.60	Yes	No	45'/750°F.	Yes	Med.	No	Rdm	No [a]
268	Yes	28	.5	19.2	2.5	.82	Yes	No	30'/750°F.	Yes	Hvy.	No	Lt.	No
269	Yes	28	.5	19.2	2.5	.82	Yes	No	30'/750°F.	No	Hvy.	No	Hvy	No
270	Yes	28	.5	19.2	2.5	.83	Yes	No	15'/750°F.	Yes	Hvy.	No	Lt.	No
271	Yes	28	.5	19.2	2.5	.82	Yes	No	15'/750°F.	No	Hvy.	No	Lt.	No
272	Yes	28	.5	19.2	4.0	1.23	Yes	No	45'/750°F.	No	v.Hvy	Med	Med	Hvy
273	Yes	28	.5	19.2	4.0	1.14	Yes	No	45'/750°F.	Yes	Hvy.	No	Lt	No
274	Yes	30	.5	19.2	4.0	1.26	Yes	No	45'/750°F.	No	v.Hvy	Lt	Med	Hvy
275	Yes	30	.5	19.2	4.0	1.23	Yes	No	45'/750°F.	Yes	Hvy.	No	Lt	No
276	Yes	30	.5	19.2	4.0	1.17	Yes	No	30'/750°F.	No	v.Hvy	No	Med	No
277	Yes	30	.5	19.2	4.0	1.14	Yes	No	30'/750°F.	No	v.Hvy	No	Med	Lt [b]
278	Yes	30	.5	19.2	4.0	1.15	Yes	No	30'/750°F.	Yes	Hvy	No	Hvy	No
279	Yes	30	.5	19.2	4.0	1.24	Yes	No	30'/750°F.	Yes	Hvy	No	Lt	No
280	Yes	30	.5	19.2	4.0	1.31	Yes	No	15'/750°F.	Yes	v.Hvy	No	Med	No
281	Yes	30	.5	19.2	4.0	1.14	Yes	No	15'/750°F.	No	v.Hvy	No	Med	No
282	Yes	28	.5	19.2	2.5	.95	Yes	No	45'/750°F.	No	Hvy.	Hvy	Med	Hvy
283	Yes	28	.5	19.2	2.5	.80	Yes	No	30'/750°F.	No	Hvy	Med	Rdm	Hvy
284	Yes	28	.5	19.2	2.5	.74	Yes	No	30'/750°F.	Yes	Lt.	No	Rdm	No

Viewed under 24X — Evidence of Cracking & Crazing

Oven Dried = 30' at 180°F.
General Appearance (naked eye) – Excellent (except #267 (blisters and uneven color) and #277 (dark blotches due to dropping hot sample onto asphalt tile.)
Color – Even dark green.
Pigment Flocculation (24X) – Heavy.

Notes:
(a) Slight gassing on the edge, blisters and poor coverage in the interior
(b) Hot piece fell onto asphalt tile.

TABLE XXVIII

Sprayed Polytetrafluoroethylene (TEFLON*) Coated Panels (Green)

Substrate – SAE 1010 low carbon cold rolled steel
Surface Preparation – vapor degrease (trichlorethylene), sandblast (#80 silica sand), oxidize (15 minutes at 715°F.)
Coating Procedure – spray (suction gun), fan dry (30 minutes at room temperature), oven dry (30 minutes at 180°F.), sinter (15 minutes at 715°F.), quench (cold water).

No.	1st coat (Primer)	1st coat Thickness mil-calc.	2nd coat (topcoat)	2nd coat Thickness mil-calc.	3rd coat (topcoat)	3rd coat Thickness mil-calc.	Final Thickness mil-calc.
300	850-204	.94	–	–	–	–	.94
301	850-204	.47	–	–	–	–	.47
302	850-204	.84	–	–	–	–	.84
303	850-204	1.01	–	–	–	–	1.01
304	850-204	.84	–	–	–	–	.84
305	850-204	.44	–	–	–	–	.44
306	850-204	.72	–	–	–	–	.72
307	850-204	.65	–	–	–	–	.65
308	850-204	.62	–	–	–	–	.62
309	850-204	.72	–	–	–	–	.72
310	850-204	.73	–	–	–	–	.73
311	850-204	.83	852-201	.26	852-201	.30	1.39
312	850-204	.85	852-201	.30	852-201	.28	1.43
313	850-204	.53	852-201	.29	–	–	.82
314	850-204	.54	852-201	.27	–	–	.81
315	850-204	.52	852-201	.28	–	–	.80
316	850-204	.48	852-201	.29	–	–	.77
317	850-204	.54	852-201	.29	–	–	.83
318	850-204	.55	852-201	.26	–	–	.81
319	850-204	.48	852-201	.35	–	–	.83
320	850-204	.55	852-201	.34	852-201	.46	1.35
321	850-204	.56	852-201	.37	852-201	.48	1.41
322	850-204	.52	852-201	.36	852-201	.45	1.33
323	850-204	.48	852-201	.37	852-201	.37	1.22
324	850-204	.49	852-201	.36	–	–	.85
325	850-204	.51	852-201	.37	852-201	.46	1.34
326	350-204	.52	852-201	.40	852-201	.35	1.27
327	850-204	.50	852-201	.34	–	–	.84
328	850-204	.48	852-201	.34	–	–	.82
329	850-204	.48	852-201	.38	852-201	.39	1.25

Finishing Materials – 850-204 Green Primer – Du Pont Finishes Division
852-201 Clear Finish – Du Pont Finishes Division

* Du Pont registered trademark

TABLE XXIX

Sprayed Polytetrafluoroethylene (TEFLON*) Coated Panels (Black)

Substrate - SAE 1010 low carbon cold rolled steel
Surface Preparation - vapor degrease (trichlorethylene), sandblast (#80 silica sand),
        oxidize (15 minutes at 715°F.)
Coating Procedure - spray (suction gun), fan dry (30 minutes at room temperature),
        oven dry (30 minutes at 180°F.), sinter (15 minutes at 715°F.),
        quench (cold water).

No.	1st coat (primer)	1st coat Thickness mil-calc	2nd coat (topcoat)	2nd coat Thickness mil-calc.	Final Thickness mil-calc.
330	850-204	.41	851-205	.52	.93
331	850-204	.40	851-205	.48	.88
332	850-204	.38	851-205	.49	.87
333	850-204	.38	851-205	.85	1.23
334	850-204	.36	851-205	1.27	1.63
335	850-204	.37	851-205	.50	.87
336	850-204	.41	851-205	.51	.92
337	850-204	.37	851-205	1.93	2.30
338	850-204	.36	851-205	.49	.85
339	850-204	.39	851-205	.82	1.21
340	850-204	.38	851-205	.40	.78
341	850-204	.37	851-205	.49	.86
343	850-204	.38	851-205	.42	.80
344	850-204	.42	851-205	1.11	1.53
345	850-204	.38	851-205	1.07	1.45
346	850-204	.37	851-205	.97	1.34
347	850-204	.35	851-205	1.02	1.37
348	850-204	.36	851-205	1.02	1.38
349	350-204	.40	851-205	.98	1.38
350	850-204	.38	851-205	.89	1.27

Finishing Material - 850-204 Green Primer - Du Pont Finishes Division
        851-205 Black Enamel - Du Pont Finishes Division

* Du Pont registered trademark.

TABLE XXX

Substrate –SAE 1010 cold rolled steel
Surface Preparation – sandblasted with #80 silica sand.
Etch –5-6 seconds at room temperature.

Bath Formula: 30.0% TEFLON 41 BX
4.0% ⌀ octanoic acid (based on the TFE weight)
15.0% FEP solids (based on the TFE weight)
3.0% carbon black solids (based on the TFE weight)
pH = 4.0

						Thick mil calc.	Fan Dried	Oven Dried	Sintering Schedule	Quench	Viewed under 26.7X Evidence of Cracking & Crazing			
											On Edge		In Interior	
No.	Etch V	A	CD	T							cracks	crazing	cracks	crazing
386	Yes 28	.50	19.2	1.6		.73	Yes	No	60/750°F.	No a	No	Lt	No	Rdm
387	Yes 28	.50	19.2	1.6		.76	Yes	No	60/750°F.	No a	No	Med	No	Lt
388	Yes 28	.50	19.2	1.6		.75	Yes	No	60/750°F.	No b	No	Med	No	Lt
389	Yes 28	.50	19.2	1.6		.79	Yes	No	60/750°F.	No b	Rdm	Med	No	Lt
390	Yes 28	.50	19.2	1.6		.75*	Yes	No	45/750°F.	No b	No	Med	No	Lt
391	Yes 28	.50	19.2	1.6		.75*	Yes	No	45/750°F.	No b	No	Med	No	Lt
392	Yes 28	.50	19.2	1.6		.75*	Yes	No	30/750°F.	No b	No	Hvy	No	Med
393	Yes 28	.50	19.2	1.6		.75*	Yes	No	30/750°F.	No b	No	Med	No	Med
394	Yes 28	.50	19.2	1.6		.75*	Yes	No	15/750°F.	No b	No	Med	No	Lt
395	Yes 28	.50	19.2	1.6		.75*	Yes	No	15/750°F.	No b	No	Med	No	Lt
396	Yes 28	.50	19.2	1.6		.73	Yes	No	45/750°F.	No	No	Med	No	Med
397	Yes 28	.50	19.2	1.6		.77	Yes	No	45/750°F.	No	No	Hvy	No	Med
398	Yes 28	.50	19.2	1.6		.73	Yes	No	30/750°F.	No	No	Hvy	No	Med
399	Yes 28	.50	19.2	1.6		.77	Yes	No	30/750°F.	No	No	Hvy	No	Med
400	Yes 28	.50	19.2	1.6		.83	Yes	No	15/750°F.	No	No	No	No	No

*Estimate

Notes:
(a) Oven shut off-door opened-cooled in oven to 450°F-removed and air cooled.
(b) Oven shut off-door opened-cooled in oven to 620°F-closed door and allowed to cool to room temperature in oven.

General Appearance – Excellent

TABLE XXXI

Bath Formula: 30.0% TEFLON 41 BX

Substrate-SAE 1010
cold rolled steel
Surface Preparation-
sandblasted with #80
silica sand.
Etch-5-6 seconds at
room temperature.

4.0% ∅ octanoic acid (based on the TFE weight)
10.0% FEP solids (based on the TFE weight)
3.0% lampblack solids (based on the TFE weight)

pH = 4.0

No.	Etch	V	A	CD	T	Thick mil calc.	Fan Dried	Oven Dried	Sintering Schedule	Quench	On Edge cracks	On Edge crazing	In Interior cracks	In Interior crazing
401	Yes	30	.5	19.2	1.6	0.82	Yes	No	60/750°F.	No a	Lt	Med	No	Med
402	Yes	30	.5	19.2	1.6	0.88	Yes	No	60/750°F.	No a	Med	Med	No	Lt
403	Yes	30	.5	19.2	1.6	0.81	Yes	No	60/750°F.	No a	Rdm	Med	No	Lt
404	Yes	30	.5	19.2	1.6	0.90	Yes	No	60/750°F.	No a	Med	Med	No	Lt
405	Yes	30	.5	19.2	1.6	0.88	Yes	No	60/750°F.	No a	Med	Med	No	Lt
406	Yes	30	.5	19.2	1.6	0.87	Yes	No	60/750°F.	No a	Lt	Med	No	Lt
407	Yes	30	.5	19.2	1.6	0.91	Yes	No	15/750°F.	No a	Med	No	No	No
408	Yes	30	.5	19.2	1.6	0.87	Yes	No	15/750°F.	No a	Lt	Rdm	No	No
409	Yes	30	.5	19.2	1.6	0.94	Yes	No	15/750°F.	No a	Lt	No	No	No
410	Yes	30	.5	19.2	1.4	0.76	Yes	No	15/750°F.	No a	Rdm	No	No	No
411	Yes	30	.5	19.2	1.4	0.70	Yes	No	60/750°F.	No	Lt	Med	No	Rdm
412	Yes	30	.5	19.2	1.4	0.68	Yes	No	60/750°F.	No	Rdm	Med	No	Rdm
413	Yes	30	.5	19.2	1.2	0.59	Yes	No	60/750°F.	No	Rdm	Lt	No	Rdm
414	Yes	30	.5	19.2	1.2	0.64	Yes	No	60/750°F.	No	Lt	Med	No	Rdm
415	Yes	30	.5	19.2	1.4	0.73	Yes	No	45/750°F.	No	Rdm	Med	No	Rdm
416	Yes	30	.5	19.2	1.5	0.77	Yes	No	45/750°F.	No	Rdm	Med	No	Rdm
417	Yes	30	.5	19.2	1.5	0.79	Yes	No	45/750°F.	No	Rdm	Med	No	Rdm
418	Yes	30	.5	19.2	1.5	0.72	Yes	No	45/750°F.	No	Rdm	Med	No	Rdm
419	Yes	30	.5	19.2	1.5	0.73	Yes	No	30/750°F.	No	No	Rdm	No	Lt
420	Yes	30	.5	19.2	1.5	0.70	Yes	No	30/750°F.	No	No	Rdm	No	Lt
421	Yes	30	.5	19.2	1.5	0.86	Yes	No	15/750°F.	No	Rdm	No	No	No
422	Yes	30	.5	19.2	1.5	0.77	Yes	No	15/750°F.	No	Rdm	No	No	No

Viewed under 27.6X — Evidence of Cracking & Crazing

Notes:
(a) Oven shut off-door opened-cooled in oven down to 620°F.-door closed-cooled
in oven to room temperature.

General Appearance-Excellent

63.

TABLE XXXII

Bath Formula: 30.0% TEFLON 41 BX

Substrate=SAE 1010
  cold rolled steel
Surface Preparation –
  sandblasted with #80
  silica sand.
Etch–5-6 seconds at
  room temperature

4.0% ∅ octanoic acid (based on the TFE weight)
3.0% carbon black solids (based on the TFE weight)
pH = 4.0

| | | | | | | Thick mil calc. | Fan Dried | Oven Dried | Sintering Schedule | Quench | Viewed under 27.6X Evidence of Cracking & Crazing | | | |
| | | | | | | | | | | | On Edge | | In Interior | |
No.	Etch	V	A	CD	T						cracks	crazing	cracks	crazing
423	Yes	28	.50	19.2	1.6	.88	Yes	No	15/700°F.	No	No	No	No	No
424	Yes	28	.50	19.2	1.6	.77	Yes	No	15/700°F.	No	No	No	No	No
425	Yes	28	.50	19.2	1.6	.78	Yes	No	15/700°F.	No	No	No	No	No
426	Yes	28	.50	19.2	1.6	.86	Yes	No	15/700°F.	No	No	No	No	No
427	Yes	28	.50	19.2	1.6	.77	Yes	No	15/700°F.	No	No	No	No	No
428	Yes	28	.50	19.2	1.6	.80*	Yes	No	120/700°F.	No	No	Med	No	Hvy b
429	Yes	28	.50	19.2	1.6	.80*	Yes	No	120/700°F.	No	No	Med	No	Hvy b
430	Yes	28	.50	19.2	1.6	.80*	Yes	No	120/700°F.	No	No	Med	No	Hvy
431	Yes	28	.50	19.2	1.6	.80*	Yes	No	120/700°F.	No	No	Lt	No	Lt
432	Yes	28	.50	19.2	1.6	.80*	Yes	No	30/700°F.	No	No	No	No	No
433	Yes	28	.50	19.2	1.6	.80*	Yes	No	30/700°F.	No	No	No	No	No
434	Yes	28	.50	19.2	1.6	.80*	Yes	No	30/700°F.	No	No	No	No	No
435	Yes	28	.50	19.2	1.6	.80*	Yes	No	30/700°F.	No	No	No	No	No
436	Yes	28	.50	19.2	1.6	.80*	Yes	No	90/700°F.	No	No	Hvy	No	Med b
437	Yes	28	.50	19.2	1.6	.80*	Yes	No	90/700°F.	No	No	Med	No	Lt b
438	No	28	.50	19.2	1.6	.80*	Yes	No	90/700°F.	No	No	Lt	No	Med a b
439	Yes	28	.50	19.2	1.6	.80*	Yes	No	90/700°F.	No	No	Med	No	Lt b
440	Yes	28	.50	19.2	1.6	.80*	Yes	No	90/700°F.	No	No	Med	No	Lt b
441	Yes	28	.50	19.2	1.6	.80*	Yes	No	60/700°F.	No	No	Med	No	Lt b
442	Yes	28	.50	19.2	1.6	.80*	Yes	No	60/700°F.	No	No	Med	No	Lt b
443	Yes	28	.50	19.2	1.6	.80*	Yes	No	60/700°F.	No	No	Med	No	Lt b
444	Yes	28	.50	19.2	1.6	.80*	Yes	No	60/700°F.	No	No	Med	No	Lt b
445	Yes	30	.45	17.3	1.6	.80*	Yes	No	30/700°F.	Yes	No	No	No	No
446	Yes	30	.45	17.3	1.6	.80*	Yes	No	30/700°F.	Yes	No	No	No	No
447	Yes	30	.45	17.3	1.6	.80*	Yes	No	30/700°F.	Yes	No	No	No	No
448	Yes	30	.45	17.3	1.6	.80*	Yes	No	180/700°F.	Yes	No	No	No	No
449	Yes	30	.45	17.3	1.6	.80*	Yes	No	180/700°F.	Yes	No	No	No	No
450	Yes	34	.50	19.2	1.6	.80*	Yes	No	180/700°F.	Yes	No	No	No	No
451	Yes	34	.50	19.2	1.6	.80*	Yes	No	60/700°F.	Yes	No	No	No	No
452	Yes	34	.50	19.2	1.6	.80*	Yes	No	60/700°F.	Yes	No	No	No	No
453	Yes	34	.50	19.2	1.6	.80*	Yes	No	60/700°F.	Yes	No	No	No	No
454	Yes	34	.50	19.2	1.6	.80*	Yes	No	120/700°F.	Yes	No	No	No	No
455	Yes	34	.50	19.2	1.6	.80*	Yes	No	120/700°F.	Yes	No	No	No	No
456	Yes	34	.50	19.2	1.6	.80*	Yes	No	90/700°F.	Yes	No	No	No	No
457	Yes	34	.50	19.2	1.6	.80*	Yes	No	90/700°F.	Yes	No	No	No	No

Notes:
  (a) Blistered
  (b) Pigment slightly flocculated

General Appearance-Excellent

*Estimate

TABLE XXXIII

Substrate-SAE 1010
cold rolled steel
Surface Preparation-
sandblasted with #80
silica sand.
Etch-5-6 seconds at
room temperature.

Bath Formula: 30.0% TEFLON 41 BX
4.0% α octanoic acid (based on the TFE weight)
10.0% FEP solids (based on the TFE weight)
pH = 4.0

No.	Etch	V	A	CD	T	Thick mil calc.	Fan Dried	Oven Dried	Sintering Schedule	Quench	On Edge cracks	On Edge crazing	In Interior cracks	In Interior crazing
285	Yes	30	.5	19.2	2.5	1.58	Yes	No	30/750°F.	No	Yes	---	No	----
286	Yes	30	.5	19.2	2.0	1.15	Yes	No	30/750°F.	No	No	--	No	----
287	Yes	30	.5	19.2	1.5	1.05	Yes	No	30/750°F.	No	Rdm	--	No	----
288	Yes	30	.5	19.2	1.0	.94	Yes	No	30/750°F.	No	No	---	No	----
289	Yes	30	.5	19.2	0.5	.66	Yes	No	30/750°F.	No	No	---	No	----
290	Yes	30	.5	19.2	0.6	.63	Yes	No	30/750°F.	No	No	---	No	----
291	Yes	30	.5	19.2	0.8	.71	Yes	No	30/750°F.	No	No	---	No	----

Bath Formula: 30.0% TEFLON 41 BX
4.0% α octanoic acid (based on the TFE weight)
10.0% FEP solids (based on the TFE weight)
3.0% carbon black solids (based on the TFE wt.)
pH = 4.0

No.	Etch	V	A	CD	T	Thick mil calc.	Fan Dried	Oven Dried	Sintering Schedule	Quench	On Edge cracks	On Edge crazing	In Interior cracks	In Interior crazing
292	Yes	30	.5	19.2	0.8	.62	Yes	No	30/750°F.	No	Rdm	Med	No	Med
293	Yes	30	.5	19.2	0.9	.57	Yes	No	30/750°F.	Yes	Rdm	No	No	No
294	Yes	30	.5	19.2	1.1	.70	Yes	No	45/750°F.	Yes	Rd m	No	No	No
295	Yes	30	.5	19.2	1.5	.73	Yes	No	45/750°F.	No	Rdm	Med	No	Med
296	Yes	30	.5	19.2	1.6	.80	Yes	No	15/750°F.	Yes	No	No	No	No
297	Yes	30	.5	19.2	1.6	.77	Yes	No	15/750°F.	No	Rdm	Med	No	Med
298	Yes	30	.5	19.2	1.6	.71	Yes	Yes	30/750°F.	No	Rdm	Med	No	Med
299	Yes	30	.5	19.2	1.6	.77	Yes	Yes	30/750°F.	Yes	No	No	No	No
351	Yes	28	.5	19.2	1.6	.77	Yes	No	45/750°F.	No	Rd m	Med	No	Med
352	Yes	28	.5	19.2	1.6	.77	Yes	No	45/750°F.	Yes	Rdm	No	No	No
353	Yes	28	.5	19.2	1.6	.73	Yes	No	30/750°F.	No	Rdm	Med	No	Med
354	Yes	28	.5	19.2	1.6	.73	Yes	No	30/750°F.	Yes	No	No	No	No
355	Yes	28	.5	19.2	1.6	.77	Yes	No	45/750°F.	Yes	Lt	No	No	No
356	Yes	28	.5	19.2	1.6	.73	Yes	No	45/750°F.	No	No	Med	No	Lt
357	Yes	28	.5	19.2	1.6	.84	Yes	No	30/750°F.	No	Lt	Med	No	Lt
358	Yes	28	.5	19.2	1.6	.78	Yes	No	30/750°F.	Yes	Lt	No	No	No
359	Yes	28	.5	19.2	1.6	.81	Yes	No	15/750°F.	Yes	Rdm	No	No	No
360	Yes	28	.5	19.2	1.6	.73	Yes	No	15/750°F.	No	Rdm	No	No	No
361	Yes	28	.5	19.2	3.2	1.16	Yes	No	15/750°F.	No	Rdm	No	No	No
362	Yes	28	.5	19.2	3.6	1.43	Yes	No	30/750°F.	No	Hvy	Hvy	Lt	Med

Viewed under 27.6X — Evidence of Cracking & Crazing

TABLE XXXIII continued

No.	Etch	V	A	CD	T	Thick mil calc.	Fan Dried	Oven Dried	Sintering Schedule	Quench	On Edge cracks	On Edge crazing	In Interior cracks	In Interior crazing
363	Yes	28	.5	19.2	3.2	1.25	Yes	No	$30/750^{O}F.$	Yes	Med	No	No	No
364	Yes	28	.5	19.2	3.2	1.29	Yes	No	$45/750^{O}F.$	No	Med	Hvy	No	Med
365	Yes	28	.5	19.2	3.0	1.16	Yes	No	$45/750^{O}F.$	Yes	Lt	No	No	No
366	Yes	28	.5	19.2	3.0	1.30	Yes	No	$15/750^{O}F.$	No	Med	No	No	No
367	Yes	28	.5	19.2	3.0	1.23	Yes	No	$15/750^{O}F.$	Yes	Med	No	No	No
368	Yes	28	.5	19.2	3.0	1.30	Yes	No	$60/750^{O}F.$	Yes	Lt	Lt	No	Lt
369	Yes	28	.5	19.2	3.0	1.24	Yes	No	$60/750^{O}F.$	No	Med	Med	No	Med
370	Yes	28	.5	19.2	3.0	1.20	Yes	No	$60/750^{O}F.$	No	Rdm	Med	No	Med
371	Yes	28	.5	19.2	3.0	1.17	Yes	No	$60/750^{O}F.$	No	Rdm	Med	No	Med
372	Yes	28	.5	19.2	3.0	1.24	Yes	No	$60/750^{O}F.$	No a	Med	Hvy	No	Med
373	Yes	28	.5	19.2	3.0	1.20	Yes	No	$60/750^{O}F.$	No	Med	Med	No	Med
374	Yes	28	.5	19.2	3.0	1.20	Yes	No	$60/750^{O}F.$	No a	Med	Med	No	Med
375	Yes	28	.5	19.2	3.0	1.38	Yes	No	$60/750^{O}F.$	Yes	Med	Med	Lt	Lt
376	Yes	28	.5	19.2	1.7	.86	Yes	No	$60/750^{O}F.$	No b	No	Rdm	No	No
377	Yes	28	.5	19.2	1.7	.80	Yes	No	$60/750^{O}F.$	No b	No	Rdm	No	No
378	Yes	28	.5	19.2	1.6	.73	Yes	No	$60/750^{O}F.$	No b	No	Rdm	No	No
379	Yes	28	.5	19.2	1.7	.84	Yes	No	$60/750^{O}F.$	No b	No	Rdm	No	No
380	Yes	28	.5	19.2	1.6	.81	Yes	No	$60/750^{O}F.$	No b	Rdm	Rdm	No	No
381	Yes	28	.5	19.2	1.6	.79	Yes	No	$60/750^{O}F.$	No b	No	Rdm	No	No
382	Yes	28	.6	23.0	1.6	.97	Yes	No	$60/750^{O}F.$	Yes c	Lt	Med	No	Lt
383	Yes	28	.5	19.2	1.6	.89	Yes	No	$60/750^{O}F.$	Yes c	No	Lt	No	Lt
384	Yes	28	.5	19.2	1.6	.74	Yes	No	$60/750^{O}F.$	Yes c	No	No	No	No
385	Yes	28	.5	19.2	1.6	.73	Yes	No	$60/750^{O}F.$	Yes c	No	Rdm	No	No

Notes:
(a) Oven shut off-door opened-cooled inside oven down to $450^{O}F.$-removed and air cooled.
(b) Oven shut off-door opened-cooled in oven down to $620^{O}F.$-door closed-cooled in oven to room temperature.
(c) Removed from oven-sprayed (2 passes) of cold water onto the hot surface-surface still hot-allowed to air cool

General Appearance-Excellent

Borden-Leben Apparatus

Friction-Adhesion-Cohesion Evaluation of General Plastics TFE Coatings

10,000 Gm Load-0.1 cm/sec-Room Temperature-100 Cycles

Code	Coating Thickness Est.	Coating Thickness Measured	Friction μ_k	Coating Solids Composition	Comments	NRL Rating
229	.63	.50	.04-.05	Electrodeposit	Sheared - Retains coverage	
233	.77	.60	.04-.05	30% TFE	Sheared - Retains coverage	
234	.81	.80	.05	10% Alumina	Extruded (Friction erratic ± .02)	IV
241	1.33	1.10	.04-.06	10% FEP 20% Chrome oxide	Extrusion and shear	
255	.85	.80	.03-.04	Electrodeposit	Extruded Lightly - Minute ruptures	
256	.77	.60	.03-.04	30% TFE	Extruded Lightly - Minute ruptures	
257	.96	.80	.03-.06	20% chrome oxide	Extruded Moderately - Minute ruptures	V
260	1.29	1.20	.03-.05		Extruded Heavily - Minute ruptures	
262	1.12	1.00	.03-.05		Extruded Moderately - Minute ruptures	
263	1.19	1.20	.03-.05		Extruded Heavily - Minute ruptures	
266	.73	.65	.04	Electrodeposit	Sheared - Retains coverage	
270	.83	.80	.04-.05	30% TFE	Sheared - Retains coverage	II
272	1.23	1.00	.02-.04	20% Chrome oxide	Extruded Heavily - Retains coverage	
273	1.14	1.10	.03-.04	20% FEP	Extruded Moderately	
305	.44	.50	.03-.04	Spray	No shear or extrusion - Excellent	
310	.73	.60	.03-.04	TFE Green Primer	Top coat shears - Coverage good	I
311	1.39	1.80	.03-.05	TFE Clear Finish	Top coat extrudes and shears	
316	.77	1.00	.03-.05		Top coat extrudes and shears	
331	.88	.90	.03-.04	Spray	Top coat extrudes-separates	
332	.89	1.10	.03-.04	TFE Green Primer	Top coat extrudes-separates	
333	1.23	1.50	.03-.04	TFE Black Top coat	Top coat extrudes-separates	III
339	1.21	1.50	.03-.04		Top coat extrudes and shears	
340	.78	1.00	.03-.04		Top coat extrudes-separates	
341	.86	1.50	.03-.04		Top coat extrudes-separates	

Note: Data supplied through the courtesy of Mr. Vincent Fitzsimmons of the Naval Research Laboratory

TABLE XXXIV

TABLE XXXV

Tabulation of Materials

MATERIAL	% Solids Content	pH	Mfg's. Designation	Manufacturer
TFE Aqueous Dispersion (stabilized with an immiscible oil rather than a surfactant)	35.0%	10	TEFLON 41BX	DuPont-Plastics Dept.
TFE Aqueous Dispersion (contains 6% TritonX-100) (based on the TFE weight)	60.0%	10	TEFLON 30	DuPont-Plastics Dept.
TFE Aqueous Dispersion (smaller particle size than TEFLON 30)	60.0%	10	TEFLON 3170	DuPont-Plastics Dept.
Green Compounded TFE Aqueous Dispersion (a primer)	39.0%	Acidic	#850-204	DuPont-Finishes Div.
Green Compounded TFE Aqueous Dispersion (High build per coat)	46.0%	Acidic	#851-224	DuPont-Finishes Div.
Clear Compounded TFE Aqueous Dispersion	48.0%	Acidic	#852-201	DuPont-Finishes Div.
Black Compounded TFE Aqueous Dispersion	41.0%	Acidic	#851-205	DuPont-Finishes Div.
FEP Aqueous Dispersion (believed to contain 6% of mixed anionic and non ionic surfactants (based on the TFE Weight)	60.0%	10	TE 9500	DuPont-Plastics Dept.
Clear Compounded FEP Aqueous Dispersion	45.0%	8-10	#856-200	DuPont-Finishes Div.
Alkyl aryl polyether alcohol	100.0%	---	Triton X100	Rohm & Haas
Sodium lauryl sulfate	100.0%	---	Duponol ME	DuPont
Sodium octadecyl sulfate	100.0%	---	Duponol L-144WDG	DuPont
Sodium Dihexyl Sulfosuccinate	80.0%	---	Aerosol MA	American Cyanamid
Sodium Diamyl Sulfosuccinate	100.0%	---	Aerosol AY	American Cyanamid
Sodium Dioctyl Sulfosuccinate	100.0%	---	Aerosol OT	American Cyanamid

TABLE XXXV - Continued

MATERIAL	% Solids Content	pH	Mfg's. Designation	Manufacturer
Highly Fluorinated compound	100.0%	Acidic	FC-128	3M Co.
Perfluorooctanoic acid	100.0%	Acidic	FC-26	3M Co.
Perfluorohexanol	100.0%	----	----	(Supplier) NRL
Free acid Fluoroalkyl phosphate plus a mixture of fluoroalcohols	100.0%	Acidic	Zonyl S-13	DuPont - Organic Chemicals Dept.
Fluorochemical Surfactant	100.0%	---	FC-95	3M Co.
Fluorochemical Surfactant	100.0%	---	FC-98	3M Co.
Perfluorooctanol	100.0%	---	---	Columbia Org. Chem Co
Omega-hydroperfluorononyl alcohol	100.0%	---	---	Columbia Org.Chem.Co
Omega-hydroperfluoroheptyl alcohol	100.0%	---	C7 Fluoroalcohol	DuPont- Organic Chemicals Dept.
Carbon Black Pigment dispersed in water	39.0%	9.0	W-7012	Harshaw Chem. Co.
Chrome Oxide Green Pigment dispersed in water	68.0%	9.0	W-6017	Harshaw Chem. Co.
Colloidal Alumina (contains absorbed acetic acid)	83.0%	Acidic	BAYMAL	DuPont- I & B Dept.
Corrosion resistant Chromium containing coating	---	---	HINAC I-X	Pennsalt Chem. Co.
Lamp Black Pigment dispersed in water	51.0%	9.0	W-7017	Harshaw Chem. Co.
Colloidal silica (alumina modified)	30.0%	9.0	Ludox AM	DuPont - Ind. & Biochemicals Dept.
Colloidal silica	30.0%	9.8	Ludox HS	DuPont
Cation exchange resin	100.0%	---	IRC-50	Rohm & Haas
Cation exchange resin	100.0%	---	IR-120	Rohm & Haas
Urea-melamines-water dil.	100.0%	---	Accobond 3810	American Cyanamid
Phenolic-water dilutable	66.0%	7.0	BRL-1100	Union Carbide
Organic titanate	---	---	Tyzor PB	DuPont-Org. Chem.
Titanium acetyl acetonate	---	---	Tyzor AA	DuPont-Org. Chem.

The above materials were modified as follows before adding to the TFE dispersion:

Perfluorooctanoic acid- a gel is made of the solid acid by the addition of a small amount of water to facilitate the addition to the dispersion

FEP dispersion (TE-9500) - reduced to 30.0% solids with deionized water and reduced to a pH of 5.0 by slurrying with an ion exchange resin (IRC-50) followed by the addition of 3.0% ∅ octanoic acid (based on the FEP weight) pH = 2.0

Colloidal silica (Ludox AM) - reduced in pH to 4.0 by the addition of 4.68% ∅ octanoic acid (based on the weight of the colloidal silica solids). pH = 4.0

<u>TABLE XXXVI</u>

<u>Terms and Abbreviations</u>

pH -- Readings using pH paper
pH_m - Readings using a pH meter
Cond - Conductivity of the plating bath in micromhos at 70^OF.

Panel (or No.) - SAE 1010 or 1020 cold rolled steel - surface area coated - .025 sq. ft.

Etch - A 5-second dip in 10% HCL at room temperature immediately preceding electrodeposition.

V   - Voltage or potential difference applied to the panel.

A   - Amperage or current applied to the panel.

CD - Anode current density in amperes per square foot. Calculated from the amperage and the surface area that was coated.

T   - Plating time in seconds.

Thickness
mil-calc. - The average thickness of the electrodeposited coating obtained by calculating from weight and area measurements and assuming the density of the TFE coating to be 2.0 gms/cc.

Final Thickness (mil) - The thickness of the film that was stripped from the panel as measured by a micrometer. This thickness is the combined thickness of the applied electrodeposit and the topcoat. The topcoat is applied to increase the strength of the deposited films so that they may be stripped without rupturing.

Fan Dried - Force dried by placing the panel in front of a fan at room temperature.

Oven Dried - Panel placed in a gravity type oven for 30 minutes at 180^OF. - unless otherwise noted.

Sintering Schedule - The time in minutes the panel was in an oven maintained at the
      or   stated temperature.
Baking Schedule

Quench - Panel placed in a bath of cold water immediately upon removal from the sintering oven.

% Weight Loss - The loss in coating weight after sintering, based on the initial fan dried weight.

Appearance (naked eye) - Refers to the general appearance of the deposit, i.e. to the
or General Appearance   presence of staining in the case of clear coatings and to the coverage or adequacy of hiding in the case of pigmented coatings, and to the presence of defects such as blisters, pits, pigment flocculation, drainage marks, streaks or roughness. Cracks are not indicated unless stated.

70.

TABLE XXXVI -- continued

Appearance of Deposit   - The same as above but viewed under 24X or 27.6X magnification.
(24X or 27.6X)

Cracks (24X or 27.6X)   - Indicates the degree of mud cracks in the coating when viewed
under a magnification of 24X or 27.6X. % values refer to the
general area of the panel covered by cracks. A value of 10-20%
normally would indicate that cracks are present only near the
edges of the panels and not in the middle areas of the panel as
the edges receive a heavier deposit and are the first areas to
exhibit cracking.

Evidence of cracking &   - A more thorough examination of the type and degree of crack-
crazing (viewed under 24X   ing. This examination attemps to differentiate between very
or 27.6X)   light surface cracking (i.e. crazing) and the deeper and wider
On edge   In Interior   cracks found, and also degree in terms of area covered (not
intensity in any one small area) For further explanation see
Page 28.

Adhesion - lbs./in.   - The force required to peel a 1-inch strip from the panel at an angle
of 90° at a speed of approximately 12 inches per minute. The
maximum and minimum values are shown. This test is conducted
on "built up" coatings.

Adhesion - Poor,Fair,Good   - Indicates the relative ease of stripping the electrodeposited
film (i.e. not a built up film) from the panel employing a
fingernail or knife.

Adhesion rating   - A more thorough method of examination than above. Increase in rating
from 1 - 5 indicates increasing difficulty of peeling a continuous strip.
Increase in rating from 5 - 10 indicates increasing difficulty in peeling
a continuous strip but decreasing resistance to removal by gouging.
Thus, ratings moving away from the ideal of "5" indicate decreasing
overall adhesion. For further explanation see Pages 25 and 26.

CCT - Critical cracking thickness.